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TRANSCRIPT OF RECORD

Supreme Court of the United States

OCTOBER TERM, 1942

No. 71

UNITED CARBON COMPANY AND UNITED CARBON COMPANY, INC., PETITIONERS,

vs.

BINNEY & SMITH COMPANY

ON WRIT OF CERTIORARI TO THE UNITED STATES CIRCUIT COURT OF APPEALS FOR THE FOURTH CIRCUIT

PETITION FOR CERTIORARI FILED APRIL 9, 1942.

CERTIORARI GRANTED MAY 11, 1942.

SUPREME COURT OF THE UNITED STATES

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[fol. 1]

IN THE DISTRICT COURT OF THE UNITED STATES FOR THE SOUTHERN DISTRICT OF WEST VIRGINIA, AT CHARLESTON, ON THE 17TH DAY OF FEBRUARY, 1941

No. 58

BINNEY & SMITH, Plaintiff,

v.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants.

Findings of Fact and Conclusions of Law

This action having been tried upon the facts by the Court without a jury, the Court doth hereby find the facts specially and state separately its conclusions of law thereon, and directs the entry of the appropriate judgment, as follows, in conformity with Rule 52(a) of the Federal Rules of Civil Procedure:

FINDINGS OF FACT

(1)

Plaintiff is the lawful owner of the patent in suit, to-wit, Wiegand and Venuto, No. 1,889,429, filed December 2, 1927, and patented November 29, 1932. This patent will hereinafter be referred to as "plaintiff's patent."

[fol. 2]

(2)

In this suit, only claims (1) and (2) of plaintiff's patent are alleged to have been infringed, and these claims are as follows:

"1. Substantially pure carbon black in the form of commercially uniform, comparatively small, rounded, smooth aggregates having a spongy or porous interior.

"2. As an article of manufacture, a pellet of approximately one-sixteenth of an inch in diameter and formed of a porous mass of substantially pure carbon black."

(3)

The agglomeration of dry powders to eliminate dustiness and facilitate commercial handling, was well known to the prior art when the application for plaintiff's patent was filed.

(4)

Claims (1) and (2) are broad article or product claims, not in any way restricted to the method taught by patentees, or even necessarily restricted to the particular product produced by their process. A product as described by these claims may result from methods quite different from that disclosed by patentees and not in any way suggested by them. In order to describe its product, plaintiff was obliged to describe it, in testimony, in functional terms. The description of plaintiff's product in claims (1) and (2) is not sufficiently definite to distinguish plaintiff's product from the product of prior art processes or from a product taught by the prior art and improved by the mechanical skill of those skilled in the art.

[fol. 3]

(5)

If claims (1) and (2) were valid, and are construed broadly, there would be infringement, as plaintiff's product and defendants' product are generally similar.

(6)

If claims (1) and (2) were valid, and are construed strictly, there would be no infringement, as there are recognizable differences between the product described by the claims of plaintiff's patent and defendants' product, such as degree of porosity, degree of purity, and degree of uniformity in size.

(7)

Plaintiff has not established that it achieved commercial success with its product described in claims (1) and (2) and manufactured by the process of plaintiff's patent. Plaintiff does not now use, and has not used since 1932, the process of the patent in suit. Plaintiff now uses, and has since 1932 used, the single liquid process described in the Glaxner Patent, No. 2,065,371 (reissue No. 21,379), for the manufacture of the pellets or beads which it manufactures and sells under the number of the patent in suit.

(8)

The product described in claims (1) and (2) of the patent in suit, did not require the exercise of the inventive faculty, and was lacking in novelty, in view of the prior art, and particularly in view of the following patents:

Lewis—1,263,082, Feb. 7, 1922;
Randall—1,405,439, Apr. 16, 1918;
Acheson—1,431,079, Oct. 3, 1922;
Knowlton and Hoffman—1,286,024, Nov. 26, 1918;
Coffin and Keen—1,561,971, Nov. 17, 1925;
Geer—1,245,700, Nov. 6, 1917; and
Trent (British)—185,430, Mar. 22, 1923.

[fol. 4] CONCLUSIONS OF LAW

(1) The patentees of the patent in suit, having originally sought broader claims than claims (1) and (2) of the patents, which broader claims were rejected, and patentees having acquiesced in such rejection, and having cancelled such claims and allowed their patent to issue with narrower claims, as disclosed by the file wrapper and contents, plaintiff is not now entitled to such a broad construction of claims (1) and (2) of its patent as to embrace the same, or substantially the same, subject matter as was contained in the rejected and cancelled claims.

(2) Claims (1) and (2) of the patent in suit, must be strictly construed.

(3) Applying a strict construction to the language of claims (1) and (2) of the patent in suit, plaintiff has not borne its burden of proving infringement, and no infringement on the part of defendants is found.

(4) Claims (1) and (2) of the patent in suit, are invalid as lacking novelty and invention.

(5) Claims (1) and (2) of the patent in suit are invalid, because they fail to define the product claimed to have been invented, in such clear, definite and exact terms as required by patent law.

An order will therefore be entered, dismissing plaintiff's action, at its costs.

A. W. Barksdale, Judge Designate.

A True Copy. Attest:

Ira H. Mottesheard, Clerk. (Seal.)

[fol. 5] IN UNITED STATES DISTRICT COURT

* * * * * * *

ORDER DISMISSING ACTION

This day again came the parties, by counsel, upon the proceedings heretofore had herein, and the Court, being now fully advised of its decision, and, for reasons set out in its Findings of Fact and Conclusions of Law this day filed as a part of the record in this action, it appearing to the Court that the plaintiff is not entitled to the relief prayed for by it in this action, it is so adjudged: it is, therefore,

Ordered

that this action be, and the same is hereby, dismissed at the costs of the plaintiff; to all of which action of the Court the plaintiff, by counsel, objected and excepted upon grounds fully stated to the Court.

And it is further

Ordered

that the Clerk of this Court certify and mail copies of this order, together with copies of the Court's Findings of Fact and Conclusions of Law, to Howard R. Klostermeyer, Esquire, Charleston, West Virginia, and Pennie, Davis, Marvin & Edmonds, 165 Broadway, New York, N. Y., attorneys for the plaintiff, and to Osman E. Swartz, Esquire, Charleston, West Virginia, and Dike, Calver & Gray, 2343 Union Guardian Building, Detroit, Michigan, attorneys for the defendants.

Enter:

(Sgd.) A. W. Barksdale, Judge Designate.

A copy, Teste: (Sgd.) Ira H. Mottesheard, Clerk.

[fol. 6] IN UNITED STATES DISTRICT COURT

Statement of Evidence

PLAINTIFF'S WITNESSES

(Plaintiff's Exhibit No. 1, filed.) [31].

TESTIMONY OF ALAN F. KITCHEL * * * [32].

* * * Direct examination * * * [32].

* * * Q. State your name, place of residence and your present occupation.

A. Alan F. Kitchel; my residence is in Old Greenwich, Connecticut; and I am president of Binney & Smith Company.

Q. The plaintiff in this suit?

A. Yes, sir.

Q. I wish you would give us a brief statement of your business career, when you got out of college, what you did then, and what positions you have held since that time, right up to date * * * [32].

* * * A. I was employed by Binney & Smith Company in 1909 as a clerk. In 1910 I was assigned to their general laboratory in charge of testing and control work on carbon black and in handling of general customer complaints. During 1911 and 1912 I spent considerable time in some of the carbon plants, becoming familiar with the product and the methods used to produce the various grades which my company sold. In 1913 I was put in the sales department in connection with the sales and introduction of carbon black into rubber compounds, and later on, some seven years later, I was put in charge of that sales work, and I have maintained that general sales supervision since that time.

I became president of the company in 1935.

Q. In your capacity, in charge of sales, and as president of the Binney & Smith Company, have you had occasion to keep in rather close touch with the rubber manufacturing industry?

A. It was quite necessary that I do that, not only in rubber work, but in the other industries that consumed carbon [fol. 7] black. I had to visit customers throughout the country and have regularly, since that time, visited their plants so that, through the years, I became familiar with their use of our material, the purposes for which they required it, the specifications and in general the changes that took place, due to the trends that occurred in the different industries, due to their own changes and also to the new grades of material we were offering.

Q. And the men who have been prominent in rubber manufacture, particularly that part which concerns the use of carbon black, are men you have known rather intimately through the years?

A. That is right.

NOTE: Numerals in brackets indicate typewritten transcript pages.

Q. Now, Mr. Kitchel, I wish you would tell the Court generally about carbon black, what it is, what its uses are and the extent of its use, etc., omitting for the present its use in rubber and only covering such general statement as will serve as a background for the further consideration of it in connection with rubber.

A. Black carbon, as distinguished from carbon black, is a pigment and goes back thousands of years, it being mentioned in Chinese history. They very largely valued it because of its use in the old Chinese inks, and the use of those inks with carbon as a base has preserved their letters to the present time. It was also responsible for the beautiful black lacquers on those pieces of furniture that came down to us from that time. However, that black carbon was essentially lamp black because it was made by the incomplete combustion of various oils. They gathered the soot from those oils and used it and lamp black made from those oils has been the principal black pigment down through the ages to the '70's when, after the discovery of natural gas in Pennsylvania, the gas was used to make a new type, gas black carbon, but being what we term carbon black. Because of its superior quality for most work the new carbon black displaced lamp black for the general arts.

Since that time lamp black has dwindled to a very great degree, whereas carbon black has grown immensely through the years.

[fol. 8] That, in general, was the beginning of carbon black, and up until the era of rubber which you mentioned, the carbon black was used principally in trades, largely paints, inks, etc., paper, stove and shoe polish and things of that nature. They valued it because it had a very good color, intensely black, to a degree unknown until that time.

By the Court:

Q. What are the characteristics of carbon black which make it superior for those purposes to lamp black?

A. As we know it now carbon black goes a great deal farther and, if you put lamp black and carbon black in a pile before your eyes and take lamp black and impose carbon black against it it would immediately look gray.

Q. By reason of other constituents contained in the lamp black other than carbon?

A. Possibly so. I would not know the answer to that.

By Mr. Edmonds:

Q. I think you might tell a little more about the general procedure incident to making carbon black, just enough to indicate what that procedure is.

A. Carbon black or, as we term it in the trade today, channel black, is made by impinging a flame of natural gas in an old fashioned lava tip, against a steel channel. That flame hits the channel and deposits soot on the channel, and that, by mechanical means, is scraped off and taken on into our plant. Just picture, if you will, what that means today. In the State of Texas there are millions of those gas flames burning in the burner houses impinged against those channels, making what we require in this country and abroad in the arts. The film which is scraped off the channel comes in rolls. You will notice the lightness of it.

Q. The receptacle which you just presented illustrates the black when it is scraped off the channel?

A. Yes.

Mr. Edmonds: We offer this in evidence as Plaintiff's Exhibit No. 2.

(Plaintiff's Exhibit No. 2, filed.)

[fol. 9] The Witness: In order to be able to handle that further we have to take certain steps. As it is light and fluffy it simply floats in the air and occupies so much space it would be bothersome to handle it in the plant and for the customer to use it; on account of the space required it would not be practical in a commercial sense to handle it in that way. So we take a step after that is scraped off the channel to remove part of the air that is entrained in it by subjecting it to agitation. After churning that mass for a white it gradually settles down. I have a specimen here of material after it has been so treated in the first step—you will notice it is much more compact.

Mr. Edmonds: I offer in evidence the receptacle just produced by the witness bearing the inscription, "carbon black as it comes out of the air separator."

(Plaintiff's Exhibit No. 3, filed.)

The Witness: From that on, the black is taken from the agitator. In the first practice of the industry, it was put into a barrel, a regular sugar barrel, which in those days

would hold 50 pounds; later it was stepped up to 75 pounds, and in the same barrel as before; and later on it became the custom to pack it into paper bags, and in those bags you could get 12½ pounds. We changed the consistency or the bulking of the black from the point where it came off the channels from two pounds per cubic foot to about twelve pounds per cubic foot. That is the state in which it was put in the bags and shipped to the customer.

Mr. Edmonds: We offer in evidence the specimen just produced by the witness as Plaintiff's Exhibit No. 4.

(Plaintiff's Exhibit No. 4, filed.)

Q. These three samples or specimens, Exhibits 2, 3 and 4, then represent the successive steps in making the carbon black products?

A. In three successive stages in the operation of the manufacture of carbon black, yes, sir.

[fol. 10] Q. Now will you proceed with the discussion of this channel black in rubber, about when it was discovered that the black is a re-enforcing agent, its use in rubber, and what the history of the development has been?

A. I would like to comment * * * [32-39]. * * * that in this form in what we now call "uncompressed black" that black was largely used in paint, etc., and still goes to those industries in that form. They want it in that form because in general in all of the trades it is used in bulk in liquid vehicles, either water or oil. It is uncompressed but it is ideal because it is easily wetted and they disperse it according to their own processes.

At that particular time, just prior to the introduction of carbon black in rubber, when I first joined our company, the total carbon black produced in the whole industry was somewhat less than 25,000,000 pounds per year, and it was practically all made in this State of West Virginia. It was valued because of those properties as called for by the trades.

You ask about rubber. It was in 1912 that my firm received its first inquiry from one of the country's leading tire manufacturers for a sample of carbon black. That led to forwarding it to them and was followed by a request for other lots during the next year. It was in 1913, as I stated before, that I was assigned to the sales work to follow up this apparent interest of the rubber manufacturer in our

carbon black. We had had no business of such nature before. It was not until somewhere late in 1913 that I was able to get a line on what was going on, because on my initial visits I found them very secretive and absolutely unwilling to divulge what was going on. Late in 1913 it was disclosed to me that this company had in mind a new step in rubber compounding which, if successful, would revolutionize the whole art of tire making as it was known at that time. Up until that time automobile tires—you probably remember them—had white treads. They were made with a canvass carcass for the structure of the tire, and that white tread was made from rubber with a zinc oxide as a reinforcing agent * * * [39-40].

[fol. 11] * * * The Witness: I would like to see samples of some raw rubber and also some zinc tread stock. That, sir, is known to the trade as smoked sheet. This is compounded with the zinc oxide. The others are simply different types of rubber used by the rubber industry.

Mr. Edmonds: We offer in evidence as Plaintiff's Exhibit No. 5 the sample smoked sheet, and as Plaintiff's Exhibit No. 6, the sample of rubber shown by the witness.

(Plaintiff's Exhibits Nos. 5 and 6, filed.)

The Witness: Those tires made with the canvass carcass and that zinc oxide white tread gave fairly balanced performance in that they were getting on the road from 3000 up to, at the highest, 5000 miles. At the end of such time the white tread would have been entirely worn off and the canvass would have been so weakened that it was not good for further use.

Then a new idea was explained to me in 1913 of improving the tire life and performance of the automobile tire being then made. The idea was to improve the carcass so it would last longer, and at the same time it would be necessary to improve the tread. They were getting at the improvement of the inner carcass by the use of what was known as cord construction. They had heavy cord which would be much stronger than the canvass. To obtain additional mileage and wearing qualities of the tread, they were embarking upon the use of carbon black as a reinforcing agent for that rubber instead of zinc oxide. They made a black tread tire instead of the white one you are looking at.

May I see a sample of the black tread stock? You can see the difference. The carbon black is in that same rubber instead of the zinc oxide.

Mr. Edmonds: I will introduce this sample of reinforcing stock with that carbon black as Plaintiff's Exhibit No. 7.

(Plaintiff's Exhibit No. 7, filed.)

[fol. 12] The Witness: As results showed later, that development of the new tire was a very sound one because we have found that, instead of the old three or four thousand mile use, it is not at all uncommon to have 15,000 or 20,000 miles' use for the black as against the white tire. Is it advisable at all to state anything about the function the black plays in that rubber?

Q. I think you might to the extent that it is known.

A. There are many theories advanced, but the one that has come to be most popular with us is the one that the carbon black serves the function of gluing together the rubber particles in that mass. A parallel case would be, it is known you can take two blocks of wood and glue them together in such a way that when force is applied the wood will fracture, not the joint where it is glued. When rubber is bonded together with carbon black in vulcanization, the bond is stronger than the rubber unit itself * * * [40-42].

* * * A. This led eventually, after trying out several different grades of black in larger and larger quantities at this tire factory, to the first contract which we made for the supply of carbon black. That was to us quite amazing because it involved in the first year a delivery of 1,000,000 pounds of black, whereas the biggest previous customer only took 500,000 pounds. It opened up to us an amazing vista in the tonnage of our product. I have mentioned the fact that in that year 1912 the general production of all carbon black was about 25,000,000 pounds. Look at what has been done in the last few years. It has got up to almost 500,000,000 pounds,—twenty times as great as before. The contract of 1,000,000 pounds has grown until the carbon black industry as a whole is shipping and delivering to rubber consumers throughout the world 400,000,000 pounds in one year. That gives you some idea of the general growth.

Mr. Edmonds: I would like to introduce in evidence at this point a copy of the bulletin to which Mr. Dike referred,

calling attention particularly to a part on the first page [fol. 13] which gives the production of carbon black in this country for a series of years from 1919 to 1929. There is a separate curve that gives the consumption and use and a third that gives the stock on hand and really all the data that one wants about the industry. I will offer in evidence a copy of the Bulletin as Plaintiff's Exhibit No. 8.

(Plaintiff's Exhibit No. 8, filed.)

Q. I wish you would proceed with the presentation of this matter of the use of carbon black in rubber manufacturing, pointing out what particular problem confronted the rubber manufacturer directly connected with the use of carbon black, what the magnitude of the problem was, what efforts were made towards solving that problem, and what success attended those efforts.

A. I might say, sir, that our success in introducing the black to other rubber customers was not quite so easy as these figures on consumption may indicate. We found that the rubber people in the use of carbon blacks brought up problems of a different nature than we had ever met before. Their specifications, for instance, for black in rubber were totally different from the ink and the paint man. He wanted color in his paint. The rubber man wanted it in a dry state. He was thinking only of reinforcing and not of color. It just happened to be black, and he did not want it black. We ran up against the situation where we knew absolutely nothing about the customers specification or how they wanted various grades. Also the fact came up after the introduction of those black tires that the users or automobile owners were very suspicious of those tires and saw a change from the clean looking white tire to a black one and the suspicion arose that that black was to mask something spurious. But by far our greatest difficulty was that the black as we had to produce it for our various customers at that time, when the rubber man took it into his plant and milled it dry and used it with this heavy resistant smoked sheet rubber, there was just chaos in the user's room. When it is incorporated [fol. 14] on the mill it rolls and boils and is sheared. When the compressed black moved along in the travel to the mills it got into the air, and at every pop there was, some of this loose black went up into the air and in the room where the work was being done and there was a cloud of this fine particle stuff sifting everywhere. The men were covered

with it, their machinery and equipment were covered with it and it got out of the windows and other parts of the factories and even into the general offices and it raised a mess that had us in hot water all the time.

I would like very much to illustrate just how black in this loose uncompressed form that we had to work with at that time looks when anybody attempts to handle it. We will safeguard it in such a way that it will not cause the trouble which it did in early use. May I have the uncompressed bag in the cage? This cover is put there only to protect us. If we did not do that it would do as it did in the factory and would fill this whole room and settle gently on everything. What I propose to do is to have Mr. Parsekian take a container of the uncompressed carbon and show what it does in the course of a very short travel between that container and the glass in the bottom of the case. What I expect to see is, if that drops out of the container we will see if any dust arises or not.

(A demonstration was then given.) See those clouds of black within that container. If that cover was not on that cage it would be all over us. We will see what happens in a very few minutes from the atmosphere of that cage.

Q. Will there be a collection of dust on the inserted sheet?

A. That will soon be evident.

Q. It is quite evident, is it not?

A. Is there anything showing on the paper?

Q. Yes, like the first layer of snow, only it is black.

A. Let us look at it, please. Will you rub your fingers around it? (A demonstration was then given.) That illus-
[fol. 15] trates it. I have another piece of paper inserted there but we will look at that later.

Q. This particular sheet was probably in there thirty seconds or a minute?

A. Certainly not more than thirty seconds. Besides that physical inconvenience there were other points as bothersome to us and to the consumer. Working for eight, or as long as ten, hours at a stretch in an atmosphere of that kind, the manufacturer was very suspicious and very fearful that might create a health hazard to his employees. Besides that the matter of dust is a very common occurrence and it was feared that the dust floating through the air might cause an explosion. Another important fact was that in the same plant where they attempted to make rubber for the tires they also manufactured other rubber goods. Some

companies would say that with all that dust floating around they could not make other colors under these conditions. Several steps were taken in the early days to get ahead of that. The manufacturers put big hoods over their mills; where this was done and they applied suction, that was drawn up and taken outside where it did no harm. Some manufacturers even went to the extent of putting up separate rooms where they did nothing but mix the black in the room. In a visit to the Goodrich plant some years later they took me to a whole building they had put up where all black compounding would be carried on and they would concentrate the nuisance that the dust created.

Some very amusing things occurred. I remember one small plant in Pennsylvania where they found it absolutely impossible to get their regular force to handle this black and they had to set up a night force which consisted of what they called the "black men." They were men that were hired at double wages to submit to going there at night, when the other workmen were not there. But the boarding houses in that town passed the "black man" from one to the other until it was a joke all over the neighborhood.

That brought us up against the fact where the rubber men would state, because of facing these problems, "Un-
[fol. 16] less you can do something about this we will try very hard indeed to develop and find something that will take its place. Let us have the material without the disadvantages connected with it." That was serious to us as there was an increasing demand. We had built up our production and if our stuff was thrown out it would leave us with unnecessary production and material which would be a drug on the market. It was a very serious matter from a financial standpoint, so we thought we had to do everything we could to find a solution for it.

That produced what has been known as the "era of dust," which lasted from 1915 to 1930. During all that time this problem was being attacked in every conceivable way by rubber men, by carbon black men and by anybody who knew the seriousness of the problems involved. In general those attempts took three rather different directions. In the first place, the rubber men themselves were up against the problem in their own plants and approaching it along different lines. The first line was on the basis of a binder which would hold the particles together. The second step, largely by rubber men themselves, was by the use of a wetting

agent, wetting that mass and driving out the air. The carbon black manufacturer in general devoted his attention to getting at the problem through what we might term "compression" * * * [43-49]. * * * The binders were wax, resin and things of that nature. The rubber man said, "I do not want those in my product. I do not want black with binders in it. I have to add my various compounds in proper order, with the proper amount, etc. and I do not want a foreign substance in the black given to me because I have to control my rubber compound from beginning to end. I do not want any foreign stuff." We found the results obtained from the use of a wetting agent did not look very successful for the reason that after it is wet down and dried to a point where the wetting agent is driven off, you get a lumping of material which is relatively quite hard. He said, "It is too hard to do its work. If I get it spread through the mix it will give me what I want, [fol. 173] but if anything interferes with getting that through the mass it is different."

I would like to mention here the work by some of the rubber men in those days, men like Geer and Randall. It was my great privilege to know Dr. Geer. I met him and had the opportunity of discussing this problem with him. He was the author of a great deal of scientific rubber literature and made many contributions.

Q. These men were among those who made some efforts towards solving this problem?

A. They made efforts over a period of time. Many of them took out patents on some of their ideas. We have reference to their patent work here. We realized in this matter of compression that the job was to get enough air out of the black, but do it in such a way as not to take it beyond the point where it could be readily used. We have recently gotten an idea of the complexity of the problem. It was very intricate and up until a short time ago we had never seen a particle of carbon black because the particle was so small that it was smaller than a wave of light and we could not see it even with a microscope. Through the use of the electron microscope we now have pictures of the carbon black particles themselves. Instead of it being fine we found it superfine because those photographs show that the diameter of the particle of our carbon black is 28 mm. or one-millionth of an inch.

By the Court:

Q. What is the shape of it?

A. Spheroid. That diameter and the fineness of our particles give a product which will spread over a surface area of 13 acres per pound. That takes me up to the point where we actually got onto different stages of compression.

Q. Before you get to that, you spoke of these expedients that were offered by men who were leaders in the rubber industry and their contributions towards a solution of this dust problem. Were any of those successful or did any of them go into use?

A. I never knew of any of them being adopted by any consumer or used at all in the manufacture or preparation [fol. 18] of carbon black. They were experimental things carried on inside of various plants.

Q. When that was demonstrated, what did the carbon black industry do?

A. We continued in our general approach along the line of compression. I would like to explain there that as to these 12½ pound bags containg fluffy black, in compression we developed various steps. The first was to take that bag and put it into either a mechanical or hydraulic press and squeeze it down to get rid of some of that air. That had to be done with extreme care because the only place for the air to get out was in the end where it was tied. That pressure had to be applied very gently and slowly. We compacted down to a flat package. That is what is now called "single pressed." In the first days it was the only step taken. When black of that nature was put on the rubber mill, it helped a little, but we found that the dust was apparently in almost as great volume as before, so we went a step further and that bag was taken into another press and squeezed down in the opposite direction, thereby reducing the cross-section of the bag. That made the bag quite compact indeed.

May I see a sample jar of the double compressed black. That, sir, is the black compressed in the two dimensions.

Mr. Edmonds: I will offer this sample of heavy compressed black in evidence as Plaintiff's Exhibit No. 9.

(Plaintiff's Exhibit No. 9, filed.)

The Witness: I would like also to show his Honor what the bulk in a package looked like in comparison with the

loose fluffy material. I have a picture which shows what it looks like after it has been compressed. It shows the lump character of the stuff.

Q. The surface of the carbon shown in the photograph is just as it exists——

A. (Interposing) It has not been touched.

Q. Just by running the fingers through the material it shows the type of the material?

A. That is right.

[fol. 19] By the Court:

Q. Is that the form which the ink manufacturers use now?

A. No, sir, he still uses the loose form.

By Mr. Edmonds:

Q. This is the form in which the rubber industry used carbon black?

A. Yes.

Mr. Edmunds: I will offer in evidence these two photographs just referred to by the witness as Plaintiff's Exhibits 10 and 10-A.

(Plaintiff's Exhibits Nos. 10 and 10-A, filed.)

The Witness: There was one additional step somewhat further in the line of compression that was taken by one or two manufacturers. This, I believe, was covered by a patent to McMahan. They had their heavy hydraulic compressors installed and they took our bags as delivered and opened up the top of this hydraulic and put our bag in and closed it and then exercised tremendous pressure. That pressure would squeeze up that loose mass of black. It destroyed the bag and when it was taken out it looked like a cheese. The paper had been destroyed but it was found, and I was told at that time, that that pressure was so great that it compacted the black to the extent where in putting it in the rubber mill they found it difficult, because of the extra time required for milling and that it would not disperse easily or thoroughly.

We had one difficulty in addition on these so-called heavy compressed or double compressed bags. When we took them into our press we naturally crimped the paper and the black was squeezed into those folds. But when we delivered the black the rubber men said, "Yes, it is not so dusty"

and he would open it and put it on his mill. They had to shake it and get the very dust we were trying to eliminate through compression * * * [49-54].

* * * Q. What was the effect of compression with respect to the dust problem?

A. I would say that it was of assistance but only a pallia- [fol. 20] tive. It did not cure it. It was bad, and continued to be bad but only to a less extent.

Q. Was that carbon black in compressed form used throughout the decade through 1920 to 1930?

A. It was the only thing that was used by the rubber customers during that period. Compressed black came to be used to a far greater part of all that which was used.

I would like to show one of the advantages there is between compressed and uncompressed black. Let me see if there is any black in that container, if it has gotten on the paper. Will you rub it? It is still there. We could leave it for the rest of the day and still find some sifting down very gently.

Q. And until tomorrow?

A. Yes. I would like for you to take some compressed black and put it through the same procedure. I think you will see the lump condition of the black will throw some down and there will be some dust with which it will have to contend.

Q. Is that double compression?

A. Yes, it is double compression. Will you put that paper on, please? (The attendant did as requested.) There is no doubt that due to the compression there are lumps of that black which will carry down without breaking up. I can see some of it has hit the bottom of the cage. There is plenty of dust and it will show the evidence on the paper without waiting at all. Look at it now, please? Is there anything on there? There it is. You can readily see from that that although it has given a little bit of help, the problem is still there. It was still with us to continue to try to do something about this dust. We must control it in some way or other. The rubber man said "we feel we want to use that material and it is doing a grand job but it has so many penalties involved that it must be given the utmost attention."

On that whole work of the various wetting agents and binders there was quite a number of patents taken out dur-

ing that period, but the compressed black was the only thing [fol. 21] I know that was used or attempted to be used throughout the industry.

By the Court:

Q. I doubt that it has any materiality, but I would really like to know briefly how they got the carbon black into the crude rubber.

A. There are people here, rubber people——

Q. (Interposing) I do not want any technical discussion. I had an idea that rubber was some kind of a liquid form and this stuff was mixed into it. I gather from what you stated I was entirely wrong.

A. Do you remember the smoke sheet? I have seen the compounding carried through from beginning to end in point of mixing form. They will weigh out so many pounds of that smoke sheet and so many pounds of the chemicals that go into it and they will have them arranged in such order they will get enough at the proper time and they are taken into the mill room. They have one mill, or a battery of them, and they are simply double rollers. One roller goes at somewhat a different speed from the other. The material is in dry form.

Q. Is it heated at all?

A. The rubber rolls, as I understand it, have to be cooled because so much heat is generated and the rubber is so tough.

Q. How does carbon get into it?

A. They put a dry chunk of rubber and add the sulphur when the rubber is there and the rolls go around and chew it. It goes off like a gun sometimes.

By Mr. Edmonds:

Q. I would like to have an opportunity to give the Court an illustration of just what that is if it will help you to visualize that operation.

The Court: Go ahead.

The Witness: Because of the fact that the new binders acted as I stated, because we knew that our own type of compression was only an approach and not an answer, and because we knew that further pressure went too far, we realized we would have to really get down to work and that [fol. 22] resulted in setting up a research department where

specialized attention could be given to just that problem and nothing else, * * * [54-57]. * * * so an answer could be derived by us. That was our next step, sir.

By Mr. Edmonds:

Q. Did you do that and with what result?

A. We did. Along in the '20's that work in a specialized group was successful and resulted in the Wiegand-Venuto patent.

Q. I wish you would state just what that new product of Wiegand-Venuto was physically and what its essential characteristics were and point out why it was that that product possessing those essential characteristics did supply the needed thing, that is, the solution of this dust problem.

A. The minute we had this product available we saw with our own testing work it would apparently hit the dust problem correctly, as we wanted to, and that it could then be put down rather concisely as to what it had done. It seemed to bear four points which were cardinal points in this whole thing. It was easy to look back and see how each of those ideas bore on the problem, and they established themselves in about this order. No. 1, the product had to be pure carbon black. We could not have binders, we could not have wetting agents that would cause trouble. No. 2 was that it had to be hard enough to withstand handling, transportation and use by the consumer, so there would be no trouble from that standpoint. No. 3, it had to be soft enough. If it was too hard it would not serve its purpose, so it had to be soft enough to get the results. No. 4 was this new point that we had not seen before, this spherical shape. Because of its spherical shape it had what we call "flow-ability". In other words, because of the rounded particles, in handling or transporting or using them they turned and rolled over and had no corners to break off and did not create dust. Those four cardinal things in this new product seemed to bring out such new and novel thoughts that we were naturally very enthusiastic about the results of our attack on this problem.

[fol. 23] I would like very much, if there is time, to illustrate all the points that I have mentioned by making some exhibits here. I would like first for you to look at some beads in the specimen jar. We call these "beads" as a matter of terminology. I think you can see the dif-

ference between the new product made by the processes that have been used and others, by an exhibit of the two-liquid process beads.

Q. This receptacle contains the product which is illustrative of the invention of the Wiegand-Venuto patent?

A. Yes, sir.

Mr. Edmonds: I will offer in evidence this receptacle of carbon black beads which is produced by the witness as Plaintiff's Exhibit No. 11.

(Plaintiff's Exhibit No. 11, filed.)

Q. This particular receptacle full of beads,—how were those beads produced?

A. They were produced by the method outlined in the patent, what we have now come to know as the "two-liquid process."

Now I would like to show photographs of a bag of beads treated in the same way as the other photographs.

Q. Is this the photograph?

A. Yes, sir.

Mr. Edmonds: I will offer the photograph identified by the witness in evidence as "Plaintiff's Exhibit No. 12" * * * [57-59].

(Plaintiff's Exhibit No. 12, filed.)

* * * A. Here are the beads.

Mr. Dike: I will ask to have it appear in the record what those beads were.

The Witness: I imagine those are our current products.

Q. By what process are those made?

A. What we call the "single liquid" method. May I see the hourglass? We have a rather neat way of showing that.

[fol. 24] Q. Would there have been any visible difference if the beads we have demonstrated here on a sheet of paper had been made by the two-liquid process?

A. No, sir, they behave in exactly the same way. This simply brings out that flow through the spherical shape. We do not want to offer that, do we?

Q. No, that need not be offered.

A. I might mention if that were uncompressed black in the top of that hourglass nothing would go through at all. May I see the two cylinders to illustrate some of this?

Mr. Edmonds: I will ask that this hourglass be marked "Plaintiff's Exhibit No. 13".

(Plaintiff's Exhibit No. 13, filed.)

The Witness: I have here two cylinders of the same size; one has a charge of heavy compressed and one a charge of beads. This is the old compressed black. You can see what happens inside.

Q. Just for the record, you inverted this tube very rapidly so the carbon black fell from one end to the other. Is it correct to say that when that was done the tube was filled with dust?

A. Yes.

Q. Will you do the same with the other tube containing the beads?

A. (A demonstration was then made.) Perfectly clean.

Mr. Edmonds: I will offer in evidence these two tubular standards that the witness has just produced and referred to, one containing the compressed black to be marked "Plaintiff's Exhibit No. 14", and the one containing the beads as "Plaintiff's Exhibit No. 15".

(Plaintiff's Exhibits Nos. 14 and 15, filed.)

Q. Would there have been any appreciable difference if they had been made by the two liquid process or the dry process?

A. I believe not. I think our most interesting or, rather, elegant way to show the ease in handling this product is to do exactly what we did with the uncompressed and [fol. 25] the compressed black except that one is not protected by surrounding it in this cage. We will be very glad to do it openly without any protection whatever.

(A demonstration was then made.)

By Mr. Smith:

Q. So that the record is clear, will you please state what beads those were?

A. Regular standard.
Q. Single liquid beads?
A. Yes.

By Mr. Edmonds:

Q. Would there be any visible difference in the product or in the action when you poured them if these had been made by the so-called two-liquid process?

A. We have done it time and time again. I have never been able to notice any difference. Here we thought we had the solution of the troubles and the product with which we could go out and do a grand job from the standpoint of supplying the customers' needs and make some nice sales ourselves. It was the only thing we had seen during this period that approached or solved the problem and the only thing during all that period that was any different from the other attempts I have mentioned.

Q. By analysis of the achievement after it was made, was it by that procedure that you came to these four essentials which you thought of as being the key to the success of the achievement?

A. Yes * * * [60-63].

* * * By Mr. Edmonds:

Q. I believe you had not quite finished your last answer before the recess, had you, Mr. Kitchel?

A. Just one or two points in connection with the patented product. I mentioned the bulk or density of the blacks as made and treated, showing the steps from the very bulky dusty black, when it came off the channel of about three pounds per cubic foot; then the initial stages of compression that brought it up to about twelve pounds per foot; then the heavy compression bringing it somewhere between twenty and twenty-five pounds per cubic foot, and in this pelleted product you got up to density there [fol. 26] of approximately twenty-seven to twenty-eight pounds per cubic foot, which reflects on the density of the material. Would it be proper to call attention to some of the points in the patent itself * * * [64]?

* * * The Court: I am not just sure that I caught the weights which you gave in your last answer.

By Mr. Edmonds:

Q. Well, let us take it in a series of steps. We have the weight per cubic foot of the material scraped from the channel, three pounds per cubic foot, is it not?

A. Approximately.

Q. Next comes the material in the agitator bin, after being agitated to drive off some of the air.

A. That is practically as dense as the material in the uncompressed form; I think very little difference.

Q. What is that?

A. Approximately twelve pounds per cubic foot.

Q. What is the weight per cubic foot for the single compressed?

A. That is twelve pounds.

Q. And the double compressed?

A. Pardon me; I made a mistake. The uncompressed is about twelve pounds per cubic foot, coming out of the bins; the single compression is approximately seventeen pounds per cubic foot, and then the double compression is somewhere between twenty and twenty-five pounds, depending upon the amount of pressure used, and then the beads or pelleted product is twenty-seven to twenty-eight.

Q. Is that matter, that higher weight per cubic foot, an item of importance commercially in connection with shipment?

A. Yes, it is. Besides the freedom from dust, for which we had originally striven, another problem is added and of very great assistance in the matter of shipping material, particularly in the foreign field. In our cost of getting this black abroad to foreign countries, where no black is made and where they depend on this country for their supply, our costs are based upon the space required on [fol. 27] the ships, ocean space, which is a cubic foot measure, and therefore these pellets, requiring so much less space, enabled us to reduce the cost of transporting from this side of the ocean to the other side, and was therefore extremely helpful.

Q. When you first started shipping abroad, the charge was on the weight, was it not?

A. I don't know about that.

Q. Until the shipping companies got on to the fact that they were not getting enough revenue?

A. I should say that is right; but I am not sure of that.

Q. Then the change occurred?

A. I think probably that is right * * * [65-67].

* * * Q. Now, you had occasion to refer, in the course of your testimony this morning, to patents that were granted to various people along prior to the patent in suit for expedients which they proposed for meeting this dust problem, and I would like to ask you if the patents granted in those years of the era of dustiness, 1915 to 1930, contained explanations of the dust problem and what it involved, which confirm what you have said here today.

A. Yes, sir.

Q. I am going to ask you now to refer to some of them, and read from them typical extracts of what you have just referred to, and I will say that if the Court cares to follow this reading, your Honor will find the patents in the book heretofore offered.

I would like to ask you first if you find in those patents general statements about this dust problem, and how much of a burden it was; and if so, give us a typical instance. You might refer first to a British patent which is at the end of the first section, 127,770, and read from that page 2, lines 3 to 6, that being the William H. Taylor patent of 1919.

A. "Prior to the date of my invention, it has been generally recognized that lamp black is invaluable for many purposes, but owing to its extremely dusty character, its use has been very much restricted since even the slightest [fol. 28] movement of the air distributes the substance in all parts of a room in which it is located."

Mr. Dike: Your Honor, we admit that black is dusty, and it is very objectionable, and I think it is entirely unnecessary to go into any long discussion of that problem or that question at all.

The Court: I understand you not only admit it, but you assert it; but I don't think it is objectionable for him to make up a record within a reasonable limitation, to show the history of and the characteristics of this product. Had you finished your answer?

The Witness: Yes.

By Mr. Edmonds:

Q. Will you turn now to the Randall patent, No. 1,384,089, the patent relating to compression, but the portion of the

specification I wish you to refer to is page 1, lines 10 to 21.

A. (Reading:) "In the packing, shipment and use of carbon black and similar materials, considerable annoyance and trouble has been experienced by the flying particles of the carbon which, as is well known, do not remain in compact form after manufacture, but are easily disposed to fly about in the air. Particularly in the manufacture and compounding of rubber, the carbon particles disperse and fly about in the workroom to the annoyance of the workmen, and detriment of any white goods that may be present."

Q. Will you turn over to the other Randall patent, 1,405,439, and read from that, page 1, lines 19 to 34?

A. (Reading:) "A pigment is usually mixed with rubber being worked by the rolls of a mill, and is usually added to the rubber in the form of a powder. In this form it exhibits particles isolated from each other by air which tends to permit particles to easily be dispersed into the atmosphere and discolor different rubber stock which may be undergoing treatment nearby. Further, the quantity of pigment required to impart desired characteristics of color, quality, etc., to the rubber, is of so great mass or bulk in powder form as to cause it to mix very slowly [fol. 29] with the rubber. Thus an abnormally long milling period is required to produce a homogeneous product."

Q. By the way, did you know this Mr. Chester J. Randall, who he was or what he was?

A. I know who he was, but I was not acquainted with him personally.

Q. Was he connected with one of the big rubber companies?

A. Yes, sir; the Naugatuck Company.

Q. And what is that company part of?

A. It is part of the——

Q. Part of the United States Rubber Company, was it not, at that time?

A. Not to my knowledge. I think it was a separate corporation at that time.

Q. Will you turn now to the Geer patent, No. 1,245,700, and read from that, page 1, lines 9 to 17?

A. (Reading) "This invention relates to the compounding of rubber in a mixing mill with ingredients such as gas-black which occurs in the form of a powder so finely divided

or impalpable that if used in a dry condition they tend to spread through the atmosphere of the mill room, causing the loss of a portion of the powder and discomfort to the operatives."

Q. Is this patentee, William C. Geer, the Mr. Geer you spoke of?

A. Yes, sir.

Q. He was head of the manufacturing and development end of the Goodrich organization, I believe?

A. Not the manufacturing end. He was head of their technical division.

Q. And was widely known in the rubber industry?

A. Widely known and recognized.

Q. A master of the industry, you might say?

A. Undoubtedly.

Q. Now, will you turn to the Knowlton patent, No. 1,286,024, and read from page 1, lines 10 to 20?

A. (Reading) "This invention relates to the art of compounding a plastic gum, such as rubber, requiring consider-[fol. 30] able mastication for its incorporation with filling materials, with substances such as gas-black (a species of lamp-black produced from natural gas) normally existing as an impalpable powder, our object being to keep the atmosphere in the vicinity of the mixing mill substantially free from the dust or powder, thus preventing waste and enhancing the comfort of the operatives."

Q. Did you know these men, Knowlton and Hoffman, the patentees of this patent?

A. Only by reputation at that time.

Q. Mr. Hoffman has been well-known in the rubber industry for the past twenty years, has he not?

A. Yes, sir.

Q. And he has been connected with the Goodrich Company through that time, has he not?

A. Through most of it.

Q. Will you turn now to the Sheppard patent, No. 1,651,733, and read from that lines 12 to 26?

A. (Reading) "Carbon pigments, such as lamp-black, gas-black and other forms of carbon black, ivory black, etc., are desirable ingredients in many compositions, because of the fact that they are fine grained, permanent and of great covering power. But in certain industries their use has led to considerable difficulties. If dust from these pig-

ments mixes with the air in proper proportion, an explosive mixture is obtained, which is highly dangerous. Moreover, this dust is liable to contaminate mixtures into which it is undesirable to have it enter. Furthermore, it has been difficult to incorporate such pigments into certain kinds of compositions."

Q. What about this man Sheppard; did you know him?

A. No, sir.

Q. Did you know who he was connected with?

A. No.

Q. Another patent, McMahan, No. 1,541,233, which I might say is a patent dealing further with compression, is the next one I desire to call your attention to. Will you please read from that, page 1, lines 10 to 19?

[fol. 31] A. (Reading) "This invention relates to the preparation of lamp black for use in compounding rubber, and has for an object to prevent dispersion of the lamp black into the atmosphere during the milling operation.

"Lamp black, while being compounded with rubber on mill rolls, permeates the atmosphere and smudges the white and other colored rubber stocks which are being milled in the same room."

Q. With whom was McMahan connected?

A. Originally with Morgan and Wright, and later with the Ajax Company in Trenton.

Q. Morgan and Wright were the United States Rubber Company?

A. Not then, but now they are.

Q. And this compression scheme was well known, was it not, and associated with his name?

A. Yes.

Q. It was known as the McMahan compressor?

A. Yes, sir.

Q. One other patent I wish you to refer to, the Lewis patent, No. 1,263,082, page 2, lines 57 to 67, beginning in the middle of a sentence where he is talking of a binder, of a material held together by a binder.

A. (Reading) "And such material will aid in reducing, if not entirely destroying the dusty character of the powder and much reduce, if not eliminate, the losses incurred by powder carried away by air currents during manufacturing processes. This effects not only a saving in the material but renders the air more healthful and gives

greater ease of manipulation of the product as well as the lessened bulk."

Q. Speaking of the various elements involved in this general nuisance of dustiness, you said one item was the loss of the product that the rubber manufacturer pays for. Which ones of these patents comment upon that loss of product?

A. Geer, Knowlton and Lewis all comment on it.

Q. Another item you spoke of was the discoloration of rubber stocks from which they wanted to make things in [fol. 32] colors other than black. Which ones of the patents comment on that portion of the dust nuisance?

A. Both the Randall patents, 1,384,089 and 1,405,439, and the Sheppard patent.

Q. Which ones of them speak of that dust in the atmosphere with reference to its effect on workers?

A. Geer speaks of it, Knowlton, Lewis and Randall in the first patent.

Q. Which ones commented on the possibility of danger through fire or explosion?

A. Only Sheppard.

Q. Some of these patents disclose the expedient or proposal of reducing or eliminating dust by putting a binder into the carbon black, and you said that was distinctly bad and gave your reasons for it, and that it was unacceptable to the rubber industry. Do any of these patents comment on that fact; that is, that binders, no matter what their kind or nature, are bad?

A. Both the Randall patents comment on that.

Q. Read the particular portions of those two patents you refer to, first Randall patent No. 1,384,089, and what I would like to have you read is on page 1, lines 21 to 33.

A. (Reading) "Various processes have been devised for compacting the carbon by the use of various binders and liquids, but these have the objection that foreign materials are introduced into the carbon and into the articles into whose composition the carbon enters. This invention has as an object to compact the carbon and thereby put it into such shape that it will not disperse and fly about when handled, and it has also as an object the compaction of carbon without the aid of binders or liquids."

Q. And the other Randall patent, No. 1,405,439, and I refer to page 1, lines 34 to 47.

A. (Reading) "The addition of an adhesive substance to the pigment during the mixing operation has been tried, for the purpose of binding together the particles of the pigment so that they cannot fly from the mill's rolls, and also to reduce the bulk of the pigment and thereby shorten [fol. 33] the mixing period. But this expedient, while partly accomplishing the purpose in view, cannot always be resorted to with impunity, for the quantity of binder usually required is so great as to materially impair the quality of and in some instances even to spoil the rubber product."

Q. I neglected to ask you about Mr. Lewis of the Lewis patent. Did you know him?

A. Yes, sir.

Q. He was, in fact, identified with your organization, was he not?

A. With an associated company, yes, sir.

Q. Did his proposal ever get anywhere commercially?

A. No, sir.

Q. The next thing I wish you would speak of, just by way of explaining it, is the process whereby beads are made in accordance with the description contained in the specification of the Wiegand patent in this suit.

A. Although these pellets may be produced in various ways, the method mentioned in this patent is what we now have come to call the two-liquid process. In simple words, it means mixing the dry carbon black with a certain quantity of water, making a slurry, then putting into that slurry some gasoline or other organic liquid, then agitating for a period of time, which brings about the formation of beads which are then taken out and dried into the product as shown in the specimen jar there. We have an apparatus here to make a practical demonstration of that, on a small scale, if you wish.

Q. It is rather remarkable, is it not, how those particles aggregate into these small beads, almost as if by magic?

A. We think it is extremely interesting, yes.

Q. I would be glad to have that demonstration made here.

A. It can be easily made here in the court room * * * [67-78].

* * * The Witness: I might say, in that connection, that this two-liquid process as originally used was an expensive one, and is not now used, due to the fact that developments in the early thirties resulted in what we now call the single-

[fol. 34] liquid process, in which the expense is much less, as patented by Glaxner in 1932.

Q. The reduction in expense is due largely to eliminating the need for the use of gasoline; is that right?

A. It requires no gasoline whatever; water only.

Q. And the gasoline that you had to use for the two-liquid process had to be very special gasoline, did it not?

A. Very high quality material; yes, sir.

Q. And expensive?

A. Yes, sir.

Q. Would it be appropriate now to take out from this cage the second sheet that was put in after the first one was taken out?

A. That was the heavy compressed black which ought not to fly quite so badly, but the paper has been in there since that was run, and may show something.

Q. Mr. Kitchel, I wish you would tell us something about the commercialization of this invention, for which a patent was applied for at the end of 1927. What was done by your organization with it from then on?

A. We found that there were elaborate preparations, rather elaborate preparations, which were necessary to be done in the attempt to commercialize this product. For the first year or two, that is, during 1928 and 1929, the material was made in a sort of semi-commercial way and——

The Court: You are referring now to the process under the Wiegand and Venuto patent?

The Witness: Yes, sir. We made in that plant sufficient quantities in order to distribute it to rubber manufacturers, not only in this country, but abroad, to distribute small samples to illustrate the points we were endeavoring to bring out in our claim, that here was the answer to the problems which they had been urging us to get after. Those first two years proved that, even though we thought we had the answer, it was not quite so easy as we imagined it would be. There appeared to be a tremendous lot of inertia by the consumers. When we look at some of the business [fol. 35] conditions which existed at that time, I think that can be commented on later, but there was resistance for several reasons, among them the following.

Most of the sizable manufacturers had, by that time, either gotten black milling concentrated into a single room, which could be protected to a great extent, or even

a single building in which nothing was done but black mixing. That concentrated the trouble. By that time also what is known as a Banbury mixer had come into use in the rubber industry. The Banbury mixer, instead of being open rolls where this rubber was milled with the black and so forth, was an enclosed or internal mixer, so that the rubber and other ingredients were put in there and the thing churned up some way or other, and it was not exposed to the air, but it could then be taken as a sort of a master mix and then put on the rolls when the black was all it it. For that reason we found that our going was slower at that time than might be expected, and in fact it was considerably easier among our foreign trade because there not only did they value the less dust in the pellet product, but they also wanted to take advantage of the saving in cost, due to the space requirements for ocean transportation.

Now, our first work, the first year or two, or two or three years, was largely taken up with getting consumers to take a small lot and run their own experiment, so as to confirm the claims which we made to them, but he had several suspicions in his mind. In the first place——

Q. (By the Court) Whom do you mean by "he"?

The Witness: The consumer, the rubber manufacturer. In the first place, he had learned that if any harsh black or over-burned black—what we term in the industry grit—got into his product, that in the working of a tire tread, if there was a particle of this harsh material in there, it caused a weakening, and gradual opening up, which led to spoilage or destruction of the thing, so that by that [fol. 36] time he had been very insistent upon his specifications regarding the absence of grit.

And so, as to these pellets, he said, "How do I know there is not some particles of grit in it?" He would reason, "I cannot test it by my ordinary methods; I cannot run it over a sieve or fine bolting operation and find out whether or not the grit is there." Of course, we could give him our word as to that, that the only thing that went into the pellet was pure amorphous carbon, and that there should not be any more fine grit in that pellet than in the black compressed or uncompressed; but that was one stumbling block.

The second was the fact that he had got to the point where he had come to find out—as I said before—that if

anything had become compacted to too great a degree it would cause him trouble because he either had to mill it too long to get it finely dispersed, or if he followed the regular milling procedure, he might run the risk of having some of that black not dispersed, and that would cause trouble as bad as grit.

The third point was that he was still very suspicious of these binders that had been used in attempts to meet this dust problem. He did not want the binders. He said, "How do I know you don't have a binder in there that I cannot find?" He had to take our word for it that there was no foreign material of any kind, that it was just pure carbon; but that accumulated into quite a long time to convince him, and to get him to run tests to confirm our arguments that that was true.

Q. Did you get somewhere with that effort in the course of time?

A. We did eventually, yes, although I want to comment upon one more point there, and that is in this two-liquid process which was more expensive, plus the fact that we thought we had a perfectly logical right to, over some fair period of years, amortize the heavy cost that we had been under in making this development, we imagined we could charge a price that was commensurate with what we thought [fol. 37] was a fair basis to meet those two points, and we asked a premium for the pelleted product. That also added to the difficulties because during the years when that was so offered we were in the depression and having troubles. The rubber men were too; competition was keen and they did not want to listen to a proposition of paying something more for any of their important products such as that was, but we began to get somewhere in the early thirties.

During 1928, for instance, only sample lots were being shipped. In 1929 we had what we call factory-size orders, test orders, and we shipped in that year some 20,000 pounds. In 1930 it grew to 164,000; in 1931, 194,000; in 1932, 281,000. Now, in 1932 we got to the single liquid process, the less expensive one, as developed by Glaxner, and there our record begins to show something different.

Q. State whether or not you continued with that premium you spoke of.

A. That was removed. We brought it down to the regular price. In 1933 we sold 800,000 pounds; in 1934, we sold

3,300,000; in 1935, 15,000,000; in 1936, 30,000,000; in 1937 we sold 44,000,000; in 1938, 53,000,000 and in 1939 it was 97,000,000. It happens that for the year 1940 over 93 per cent. of our total deliveries of carbon black to rubber manufacturers was in the pellet form. Now, in this sales work, although some of the black in the pellet form is still taken in bags, a method has come about which we think is a direct result of this Wiegand and Venuto patent, and that is what is known as bulk handling.

I mentioned one of the four cardinal points that we come to look upon, this matter of the flowability. That is the basic reason for making bulk handling possible. It was soon apparent that if we in our manufacturing plants could transport and move about and store these pellets in bulk, there was no reason why the consumer could not do exactly the same thing. The final result was that many plants today among the rubber manufacturers are equipped [fol. 38]. for this bulk handling, and instead of packing these beads in paper bags, we dropped them directly into a tank or hopper car built especially for that purpose. Those cars are moved on the railroad to the plant of the consumer. He puts them on his siding, opens up the car, the beads flow out by gravity, are conveyed to the storage tank overhead above the rooms or places in which he uses it, and then it is conveyed from there to the point where it is used and nobody touches it. There is no handling of bags from the time it gets to the plant until it arrives at the point where it is used.

Q. Was anything approximating that possible with the prior forms of carbon black?

A. No, sir. It is only a product that would have what we term this flowability that could possibly meet the requirements of such handling; and I would like to say there that a visit to a modern rubber plant presents a most striking comparison over these conditions I have described in the early days. Go into one of these modern plants and some of them are painted up and are as clean as any operation not requiring black of any nature whatever. All you can see are conveyors going up to a tank and conveyors coming down from a tank. It is even weighed automatically. You don't have to weigh it by hand. It is weighed in transit from the tank to the place where it is going to be incorporated.

Now, some plants, either because they have not been modernized or because they are small, still take this pelleted black in the paper bags, but instead of 12½ pounds they get 25 pounds per bag. Those bags are loaded in the car and sent forward. The consumer takes them out and when he comes to use them, however, he simply unties the bags and it flows out, no dust, and so he does not have to use the old precautions for he can use them any place he wants. I would like right there, if possible, to show this booklet which we published some time ago, entitled "Flow Carbon," which has some diagrams of installations, both small and [fol. 39] large, in connection with where this bulk handling is carried out and exactly how the black is used.

Q. Could we take those two pages out of the book you refer to and put them in evidence? Would it be all right to remove them from the booklet?

A. Yes.

Q. (After removing two pages from the booklet.) Mr. Kitchel, you might explain what is shown on those pages and then we will offer them in evidence.

Mr. Dike: I think you might as well put the whole book in. We are going to ask him about it.

Mr. Edmonds: Well, I wish you had told me that before I removed them from the book.

Q. As I understand, this is typical of a modern rubber plant installation?

A. In general, yes. Of course, each plant has its own local conditions and way of doing it, but in general it is all the same.

(Plaintiff's Exhibit No. 16, filed.)

The Witness: I might also say, besides our own publication, there are some very interesting comments from the Government bulletins for certain years which I would also like to read * * * [78-88].

* * * Mr. Edmonds: * * * [89]. * * * I will now offer in evidence a copy of the year book for the year 1934, one of the series that includes the one Mr. Dike presented, and ask the witness to read a single paragraph on page 750.

A. That is the bulletin of 1934 but reviews the year 1933?

Q. Yes, that is right.

A. (Reading) "Although manufacturing technique showed little change in terms of yields there was one development which may revolutionize transportation methods. Increasing quantities of black were produced in the form of small, round pellets which can be transported in bulk instead of by more costly method of compressing in bags."

[fol. 40] Mr. Edmonds: We offer in evidence a copy of the year book for 1934, as Plaintiff's Exhibit No. 17.

(Plaintiff's Exhibit No. 17, filed.)

Q. The next one in the series is the year book for 1935, reviewing the year 1934, and I will ask you to read from that a short paragraph on page 836 * * * [89-90].

* * * A. (Reading) "In the carbon black chapter of minerals year book, 1934, mention was made of the development of dustless carbon black in the shape of small pellets for transportation in bulk. Information from the trade indicates that production of this type of carbon black increased materially in 1934."

Mr. Edmonds: We offer in evidence this year book for 1935, as Plaintiff's Exhibit No. 18 * * * [90].

* * * (Plaintiff's Exhibit No. 18, filed.)

Q. Mr. Kitchel, here is the year book for 1939, reviewing the year 1938. I will ask you to read a short paragraph on page 4.

A. (Reading) "Research on carbon black by producers and consumers was perhaps intensified in 1938. Much of the research resulted in narrowing the specifications for uses of carbon black in rubber. The relative proportion of dustless production continued to increase rapidly and a collateral development, shipments in bulk, grew as fast as finances would permit."

Mr. Edmonds: We offer in evidence the year book for 1939 * * * [90-91].

* * * (Plaintiff's Exhibit No. 19, filed.)

Q. Plaintiff's Exhibit No. 8 heretofore offered in evidence contains a paragraph on page 41 which I wish you would read into the record, Mr. Kitchel.

A. Which one is that?

Q. That is the year book for 1940, reviewing the year 1939.

A. (Reading) "Despite the low price level research in the fundamentals of carbon black production and consump-
[fol. 41] tion continued, as it is quite generally recognized that the behavior of different blacks under varying conditions is a fertile field for experiment. One of the products of this research (dustless carbon black) increased steadily in popularity in 1939. It is doubtful if more than 10 per cent of the present output is of the old non-granular type."

Q. In connection with the statement about the commercialization of the Wiegand and Venuto invention, I meant to ask you to refer to three of the items in this exhibit book, the first three in the last section of publications. Will you state briefly what they are?

A. They show our announcements in the recognized trade journal of that time in regard to rubber.

Q. What is the date of the first one of those several announcements?

A. November 1, 1930, in The India Rubber World.

Q. And that is in this article headed, "Dustless Micronex"?

A. Yes, that is right.

Q. And what is the second and its date?

A. From the same publication, under date of May 1, 1933, is the second.

Q. And under that same head of "Dustless Micronex," I believe?

A. Yes.

Q. Micronex is the trade name under which your company sells its carbon black, is it not?

A. Yes.

Q. And the third?

A. From the same publication, under date of August 1, 1934.

Q. And under the same title?

A. No, this is under a different title. Here we have "Micronex Beads" instead of "Dustless Micronex".

Q. Mr. Kitchel, I would now like to ask you if it is a fact that this Wiegand and Venuto invention provided anything in the nature of a new starting point for inventive activities and, if so, how that disclosed itself.

[fol. 42] A. Due to the fact that new methods for making

this pelleted product were brought forward. We have seen new pellet structures mentioned and new machines outlined which did in effect bring about the same general pelleted product.

Q. All of these new proposals flowing directly from this contribution of Wiegand and Venuto?

A. The products certainly appeared to be the same. It was only a different way of getting at that product. The fact that stuck out in our minds was the fact that certainly up until the time of the introduction of this product by ourselves we had seen nothing offered to, or used by, consumers of this nature.

Q. And were patents applied for and obtained for these further proposals emanating from the Wiegand and Venuto?

A. I believe there is quite a list of patents of that time on these various methods.

Q. I am now going to ask you to read from the specifications of some of those patents, and copies of the ones to which I will call your attention are in the book that is before the Court, the first one being Re-issue Patent No. 21,390, which was granted to this defendant, United Carbon Company, some time along in the thirties, long after the Wiegand and Venuto patent was issued. Turn to that patent and read the portion that you find on page 1, column 1, lines 1 to 16, and you can make any comment on that which seems appropriate, Mr. Kitchel.

A. (Reading) "The present invention relates to the production of agglomerates or shaped masses of carbon black, for the purpose of overcoming some of the objectionable properties of raw carbon black, such as the formation of dust, during the handling of the same, during the mixing of the same with rubber or other materials, and for producing a product which is easier to handle. As is well known, carbon black in its original form is a powdery or more or less lumpy material constituted of extremely small particles, so that during the handling of the same, air currents [fol. 43] may blow the same about to some extent, and the object of the present invention is to produce agglomerates which will be free from the said objection."

Q. That is an object that had already been attained, had it not?

A. Yes, sir.

Q. Now will you look at the Price patent, No. 2,127,137, and read the portion on page 1, column 1, lines 14 to 24, please, Mr. Kitchel?

A. (Reading) "A well recognized difficulty in handling all of these fine powders is that they sift through the containers used to confine them. Moreover, when packed in barrels or paper sacks, an excessive dusting occurs when the products are being used or mixed with other ingredients, and as a result the area surrounding the point or points of handling is filled with dust which settles on surrounding objects, contaminates other products, and pollutes the air which those working with the product have to breathe."

Q. Continue on page 2, column 1, lines 11 to 24, if you please.

A. (Reading) "Often the black is compressed in bags or other containers which become wrinkled and crimped during the pressure treatment, thus trapping a considerable quantity of carbon black in the wrinkles, and this entrapped black can only be removed by violent shaking and then only at the expense of a cloud of dust. It is obvious that the application of heavy pressures involves heavy and expensive machinery, a large amount of power, increased expense and delay in preparing the product for shipment, while the product is very likely to be caked so hard that it is difficult to disperse or to mix it with other material."

Q. Still harping on the same old things that the prior art patents harped on, is it not?

A. Yes, sir.

Q. But meanwhile a complete remedy had been provided, had it not?

A. Yes, sir; the Wiegand and Venuto certainly met that.

Q. Now, you might continue with more from that patent, page 2, column 2, lines 3 to 15.

[fol. 44] A. (Reading) "A further object is to provide an improved apparatus for making a product which is chemically the same as the original finely divided material, but which has an increased density, and is in the form of granules or pellets which are soft, substantially dustless, easy to handle and capable of being readily dispersed in or intermixed with the material with which it is customarily used, yet which has bulk characteristics which enable it to flow, as through chutes and conveyors, and to be trans-

ported in tank cars or tank trucks without material disintegration."

Q. Strikingly similar to the description of the Wiegand and Venuto product in the patent, is it not?

A. It strikes us as exactly similar.

Q. Will you please turn to Patent No. 2,120,540 and read page 1, column 2, lines 13 to 31.

A. (Reading) "The novel product thus produced, which may preferably be of substantially spherical granular shape, possesses striking commercial advantages. In the first place, it is substantially dustless in that most of its particles are agglomerated into granules of appreciable size and substantial density. In the second place, the non-adherent character of the surfaces of the granules reduces the apparent viscosity of the mass, which viscosity may be further reduced by the preferred spherical form of the granules, permitting the carbon black to be conveyed through pipes by gravity or by streams of air, to be transported in tank cars and to be stored in bulk. Beside being much more cleanly for the consumer to handle, the novel product when mixed with other ingredients disperses and diffuses more completely and perfectly in various compounded materials than commercial carbon black of forms heretofore known."

Q. I will ask you to refer now to page 2, column 2, lines 49 and 51, which is in the middle of a sentence on that page.

A. (Reading) "* * * present the striking characteristics of dustlessness, high density and non-adherent, free flowing contact with each other."

[fol. 45] Q. Does all that description you have read apply to the Wiegand and Venuto product?

A. It certain-y does.

Q. And do you agree with what he states there, that that product has striking commercial advantages?

A. Undoubtedly, sir.

Q. Now, in his description, coming along in 1932, he refers to it as novel. Was that proper?

A. It did not strike us as anything new or novel there; just exactly the same as the patented product.

Q. Will you look at the next patent, No. 2,120,541, and read from page 4, column 1, lines 15 to 25?

A. (Reading) "It is believed, however, that never heretofore has flocculent carbon black been progressively built

up in a dry state, without assistance of binders, solvents, liquids, tars or other additions, either temporarily or permanently present in such a manner as to transform flocculent carbon black into substantially dustless tenacious granules, the particles of which are united only by their own cohesive force with sufficient tenacity of structure to resist disintegration when transported in bulk."

Q. All that description applies to the Wiegand and Venuto product, does it not?

A. The product itself yes.

Q. He says it is the first time it has been made in a dry state, so his novelty lay solely in the procedure of making the Wiegand and Venuto product?

A. It would be only a method of making it.

Q. Will you please look at Re-issue Patent No. 19,750, and read from page 1, column 2, lines 10 to 22?

A. (Reading) "Thus the carbon black may be converted into small spherical granules of apparent density in bulk higher than before such treatment, of relatively tenacious structure, with each of such granules presenting a more or less polished, non-adherent surface. Carbon black in this spherical grain form possesses all of the advantages of con- [fol. 46] venience in handling and distribution above discussed and, in addition, it disperses perfectly in rubber mixtures, and is otherwise as useful or more useful in rubber making and other industries than the compressed carbon black of commerce formerly employed."

Q. Does all that apply to the Wiegand and Venuto product which had preceded it by some years?

A. Yes, sir.

Q. Go further with that patent and read the portion of page 1, column 1, lines 22 to 37, please.

A. (Reading) "The agglomerates formed by our process are of greater apparent density in bulk than the loose powders from which they are formed and consist of more or less spherical masses having such a high ratio of weight to surface that they are not dusty. Also, they have smooth, polished, non-adherent surfaces and compact, adhesive internal structures. These characteristics give the resulting agglomerates a free-flowing nature which materially simplifies handling in bulk, homogeneous mixing with other material, delivery by gravity through chutes and pipe lines, packaging in any desired weight, and all of the other con-

veniences imparted to the product by its extremes fluidity and dustlessness."

Q. Are those the same advantages that are described in the Wiegand and Venuto patent as being advantages of the Wiegand and Venuto product?

A. Aside from the difference in words, it is.

Q. One more is Patent No. 2,039,766, page 1, column 1, lines 6 to 16.

A. (Reading) "This extreme degree of fineness carries the inevitable and generally undesirable concomitant of dustiness, and this dustiness, arising from the minute weight of the individual particle, may lead to serious undesirable commercial consequences; for example, contamination of other nearby products and processes, excessive deterioration of fixtures, machinery and furnishings and respiratory and other occupational disorders in the workmen exposed to the dust."

[fol. 47] Q. Was there anything new about that in 1933?

A. It was one of the dangers we had recognized and were trying to overcome in the original work.

Q. And did overcome?

A. Yes.

Q. The dangers are the dangers mentioned in the patents of the prior art where the people tried to overcome them and failed, are they not?

A. That is right.

Mr. Edmonds: Your Honor, I wish to state at this point that I think it will be of interest and assistance to your Honor to witness a demonstration of just what this procedure is of aggregating this fine powder into these pellets, and we are now prepared to give a demonstration here that will take only a few minutes.

The Court: All right, you may proceed.

Mr. Edmonds: The statement describing the demonstration will be made by Mr. Kitchel's assistant, Mr. Parsekian, who will conduct the demonstration.

Mr. Parsekian: I have now weighed out 12 grams of uncompressed black. I have measured out approximately 145 cc. of water, in other words, 145 grams. The first thing we do is to pour the black into the water. Then we mix the black with the water. Now I weigh into there the required amount of gasoline, in this case I am weighing

out 17 grams, which is within the scope of the patent. I have here some gasoline and this is the process of weighing it. Now I just mix it up.

Mr. Edmonds: Let the record show that at this point the operator put the tube in a rotating frame and started the motor, which rotates it, and continued the rotation for what time, Mr. Parsekian?

Mr. Parsekian: The longer you continue the rotation the smoother the aggregates are.

Mr. Edmonds: How long do you propose to continue this?

Mr. Parsekian: For half an hour. I can show you the pellets in about 10 minutes, I think • • • [91-103].

[fol. 48] • • • Q. Mr. Kitchel, I wanted to ask you one other thing. Have rights under this Wiegand-Venuto patent been conferred on others?

A. Yes, sir, the Thermatomic Carbon Company, which I believe is owned by Commercial Solvents, has taken a license from us under this patent.

Q. In connection with the comment made this morning by counsel for the defendant, is there anything further in that connection you wish to say?

A. I got the intimation that it might be difficult for any other producer of carbon black to get rights under this patent. That is certainly not so. We will be glad to negotiate a license to anyone at any time.

Mr. Edmonds: That completes the direct examination of this witness.

Cross-examination • • • [103-104].

• • • Q. In your direct examination, Mr. Kitchel, you have referred several times to the Wiegand-Venuto patent. Can you give any further description of the product which you have referred to in your use of that term?

A. Which one, the original or the present?

Q. The product which you have referred to as the Wiegand-Venuto product in your direct testimony.

A. I referred to a product and two methods of obtaining that product.

Q. Will you please state whether or not there is any difference in the products which are derived from those two different methods

A. No, no difference. It is all of a very minor nature.

Q. Is there any difference in the size of the pellets?

A. There is; one can be varied at will.

Q. How is that effected?

A. I do not run the process and I do not know the makeup of the process. The idea and my understanding is that the size of the pellets may be varied in either process by varying the amount of the liquid used and the amount of agitation, etc.

[fol. 49] Q. How would you say that the two products compare? By the two products I mean the micronex or the single process, and the other, as to their internal structure.

A. I know very little about internal structure. The only description I can give is that they are both porous.

Q. How do the two products compare as to purity of the carbon black as to the single liquid or the two-liquid?

A. I know of no difference in purity.

Q. During your direct examination you discussed the question of the problem of dust around the rubber mills, and I believe your direct examination considering that problem began about 1913, is that correct?

A. 1913 was the very beginning of the use by one firm only. It took about two years for that use to become spread through other consumers, so I would say that our problem of dust became accentuated about 1915.

Q. What type of mill was used at that time?

A. In milling rubber?

Q. Yes.

A. Open roll mills.

Q. Similar to the open roll mill that was used in the moving picture a short time ago?

A. I did not see that movie.

Q. At the present time I believe you stated that there had been some change made by the rubber company in the milling of their black into the rubber?

A. Yes.

Q. Will you please describe briefly what that change consisted in?

A. This is a layman's description entirely.

Q. That is what we want.

A. The change was primarily, instead of using dry rubber and dry materials on the open roll mills, they went to the use of the Banbury mixers and the charge of rubber

is put in there and the door closed and it grinds out the mix and most of the effect is confined in that mixer.

Q. Do I understand you that the Banbury mixer is a closed mixer as compared to the open mill or mixer used [fol. 50] previously?

A. That would be a common method of describing it.

Q. Going back again to this matter of the Banbury mixer, do you know approximately when the rubber companies began the use of the Banbury mixers?

A. I am not certain. I remember seeing them in use somewhere along in the early '30's, according to my best recollection.

Q. From the patents that you have read in your direct examination there is a considerable discussion of the different so-called problems in connection with the dusty character of the carbon black; one was its explosive properties in a mill room. I believe that is referred to in the Sheppard patent. Do you have any personal knowledge of there being any explosion in a mill from the use of dusty carbon black?

A. I never knew of such an occurrence, no, sir.

Q. In connection with the statement in several of the patents that the dusty character of the carbon black affected the health of the workmen, do you know of any instance where the dusty character of the carbon black adversely affected the health of the workmen?

A. I would say that was largely a matter of fear upon the part of the users and that we never found anything of that sort from a health standpoint.

Q. In connection with this "problem," as you have termed it, of the dispersion of the carbon black in the rubber, do you know whether that difficulty exists in the use of the Banbury mixer in connection with carbon black that is wetted by a single liquid such as is described in this patent?

A. I did not get the question.

Q. I will rephrase the question. In your direct examination one point which you mentioned was that there was a problem of dispersing the hard particles of carbon black into rubber stock. I believe you discussed that in connection with the heavy compressed blacks. I believe you read a portion of the Wiegand-Venuto patent specification which discussed this problem of dispersion of hard particles

in rubber stock. The question I am asking you is this: [fol. 51] Whether those problems of securing dispersion related to the old method of milling on the open mill, or did it relate to the method of milling in the Banbury mill?

A. The Banbury was not in use when those previous forms of black had been tried. There was the heavy compressed and hydraulic plan,—the Lewis plan which dried into cake form. I would not know how to answer the question because there were no Banbury's available to try it on.

Q. Since the introduction of the Banbury mill in the rubber trade have you heard of any difficulty in securing the dispersion of carbon black which has been rendered dustless by wetting with water?

A. I have every reason to believe that the same difficulties would exist in getting proper dispersion by the use of a Banbury and a mill or a mill alone as the carbon black in any form would have to be of such type and grade as would disperse properly through a Banbury and a mill or a mill alone.

Q. In your last answer you refer to the type and grade of black as having effect on its dispersion. Would you care to elaborate on that?

A. That is a highly technical subject. There are men here who can do that.

Q. What do you mean?

A. Certain blacks under certain specifications have to be furnished for different uses.

Q. You do not refer to the question of whether it is in a dustless form or in the old-fashioned dusty or compressed form?

A. Unless proper dispersion is obtained in any form used, there is trouble in the finished product. What I want to bring out right along is that any method in an attempt to solve this dust problem that carried it so far as to make it difficult renders it dangerous.

Q. Is not the way you had of keeping away from it this two-liquid process of Wiegand-Venuto?

A. It certainly proved to be so.

Q. What other way do you have of treating this problem of forming too hard particles?

A. I was not directly connected with that work so I would not know how to answer that.

[fol. 52] Q. Do you know of any other method employed by your company to get away from it?

A. Undoubtedly there is a very fine balance between the materials used and the time element involved and the handling of same. It must be a very fine limit to secure those necessary elements.

Q. I gathered from your direct testimony, Mr. Kitchel, that there is some relation between the density of the carbon black and its dusting properties; am I correct in that?

A. Referring to the difference between uncompressed black and compressed black?

Q. Yes.

A. Undoubtedly.

Q. What about what we would call the light compressed as compared to the heavy compressed black; was the heavy more or less dusty than the other?

A. The heavy was somewhat less dusty but only a very minor difference.

Q. In your direct examination, Mr. Kitchel, in connection with your testimony and what you termed the essential characteristics of the product of the Wiegand-Venuto patent, you discussed the importance of the pellet being a pure carbon black. Do you have a copy of the patent? Will you please refer to page 2, column 2, line 100 through 105, and state whether or not this statement of the patent is what you had in mind when you specified that one of the characteristics of the Wiegand-Venuto pellet was the purity of the carbon black?

A. The reference in those lines is not to impurities that I desired to make mention of. The impurities of which I was speaking were the binders and the additional things added in some of the prior work. This reference is to some small particles of grit.

Q. The matter of purification which you have referred to is something different to that contained in the patent, is that right?

A. The point I wanted to bring out was that, realizing it was necessary to offer to the consumer pure carbon black, this product of the patent enabled us to do that without the addition of anything not wanted.

[fol. 53] Q. In connection with the next characteristic, which I believe you named in this order, was that it was hard enough to ship, as being one of the characteristics of this Wiegand-Venuto pellet. Did you have in mind that

there was some relationship of hardness of the pellets to the shipping and handling of the pellets when you used that statement?

A. Certainly if the pellets were not hard enough to stand the handling it was necessary to give them at the carbon black plant to put them up for shipment or to stand the transportation after shipment.

Q. Did you have any yardstick or similar measurement by which you could measure the hardness of the pellet to determine when they were hard enough to stand shipment?

A. I was not connected with the work so I would not know.

Q. You do not know of your own knowledge and cannot determine that from any facts you have given us?

A. I would say practical experience would be a very good test.

Q. Can you give that from practical experience?

A. I cannot, having nothing to do with that.

Q. The next characteristic you mentioned as one of the characteristics of the Wiegand-Venuto product was that it was soft enough to disperse readily. Is there any test of that softness that you can give us to help us understand a little more what you had in mind?

A. I think again the gauge would be the point beyond which the rubber manufacturer could not secure proper dispersion. I know of no yardstick in that connection, no.

Q. If you will refer to the Wiegand-Venuto patent again, please, I should like for you to refer to page 1, column 2, beginning about line 85, to the statement which reads, "and which at the same time was easily crushed by moderate pressure, as between the fingers, or by the pressures commonly employed in the rolls of rubber compounding machinery, printers' ink mixers and the like." Would you [fol. 54] say that was or was not an accurate kind of test to determine whether or not a pellet possessed this characteristic of being soft enough?

A. I have personally felt that the finger test is a very good kind. If our pellets are sufficiently soft so they can be smoothed out on paper by just the pressure of the finger and showing no harshness, I feel that would be capable of satisfactory dispersion provided such a pellet would be hard enough to ship and handle readily.

Q. One characteristic of the Wiegand-Venuto product which you described in your direct examination was its

novel spherical shape,—I believe you termed it. Do you know of any instance where the process of the Wiegand-Venuto patent was employed to produce other than spherical shapes of the agglomerate carbon black?

A. If that process was used to produce agglomerates of other types?

Q. Yes.

A. My answer would only be by hearsay, that is, from reports in connection with work going on at the time. Any number of things have been tried with any number of results. As to shapes coming out of there, I would not know as to that possibility.

Q. If you will refer to the Wiegand-Venuto patent again, please, on page 1, column 2, beginning at line 95, I am reading from the patent: "While the pellet form is a very convenient form of the carbon black, the shape of the particles is not the most important characteristic of this novel carbon black." As I understand from your direct testimony, you disagree somewhat with that statement in the patent, in view of your statement of the fourth characteristic of the purpose of these pellets?

A. I was commenting entirely upon the pellet form of the product. I am so badly versed in matters of patent wording that I confess I am totally at a loss on that.

Q. There is not much question, is there, Mr. Kitchel, but what that portion of the patent I have just read to you is rather clear, that at least the patentee did not consider the [fol. 55] pellet form was the most important factor of their novel product; would you say that?

A. I have no way of knowing what was in their minds at all.

Q. Judging from the language of the patent yourself what would you say about it as an ordinary business man?

A. It says, "the shape of the particle is not the most important characteristic of this novel carbon black."

Q. You have in your direct testimony given four characteristics which you stated as being important characteristics of this Wiegand-Venuto pellet. Would you say which of those characteristics you consider, as a business man, the most important characteristic of that product?

A. I would say that either one alone would have been of very little use. It was the proper combination of those things that seemed to make it of such great value to us.

Q. Then you would say, would you, that the thing that solved this problem of dustiness in the rubber companies that you have referred to was, first of all, a pellet of pure carbon black which was sufficiently hard to be handled and which was sufficiently hard so that it could be shipped without disintegration and yet so it would be soft enough or break down under slight pressure? Is that in effect a description of the product to which you have referred in your direct testimony?

A. That combination plus the spherical form.

Q. In connection with the flowing of the carbon black pellets, which is one of the matters you have mentioned and concerning which you testified on direct examination, I understand that it is the shape of the dustless carbon black which gives you the flowable characteristic of the black, is that correct?

A. I understand the shape of the spherical pellet that rolls upon itself makes that flowability.

Q. If we had a grain similar to a grain of sand in the hourglass instead of this spherical pellet, would you say [fol. 56] that material also possessed this characteristic of flowability?

A. Sand is certainly flowable.

Q. What about granulated sugar?

A. To a degree. Any of those things which have sufficient roundness would provide a degree of flowiness.

Q. So if carbon black were in the same granulated form we would expect it would flow?

A. But it would not remain dustless because if it had any roughness in shape, in handling those corners would be knocked off and handling would create dust by so doing.

Q. In your direct examination you have stated on several occasions that the beads made by the single liquid process and the beads made by the two-liquid process possessed no visible differences, is that correct?

A. Yes.

By the Court:

Q. Did not you go further than that and say that as far as you knew there was no actual difference between them?

A. That is the question.

By Mr. Smith:

Q. Upon what did you base that observation, Mr. Kitchel?
A. Just visualization.
Q. Just as we might look at that here?
A. Yes.
Q. That is not based on any scientific or microscopic test with which you are familiar?
A. Not that I have done myself; that information is available.
Q. In connection with your direct examination you referred to the Godfrey L. Cabot patents. Are you familiar with any product which is on the market which is similar to the product mentioned in those patents? • • • [104-116].
• • • A. I am familiar with the product offered by the Cabot Company in the form of a pelleted black.
Q. What is the trade name of it?
A. Spheron.
Q. When did you first know of that product? • • • [117].
[fol. 57] • • • A. To the best of my recollection, whether it may have been designated as "Spheron," we met a pelleted black from that source some time around 1933.
Q. Prior to 1933, as I understood your direct testimony, your company had introduced to the trade pellets which were made under the two-liquid process, is that correct?
A. At first.
Q. I believe you stated that covered the production for the years 1930, 1931 and 1932, am I correct in that?
A. 1929, 1930 and 1931.
Q. How much of the product was put on the market in 1928?
A. Only very small sample lots.
Q. Would you give them again?

The Court: They are in the direct testimony.

A. I have them here. In 1928 it was a very small amount, probably less than 5,000 pounds.
Q. And in 1929?
A. About 20,000 pounds.
Q. Do you have the figures for the other years?

The Court: Give me the figures for the other years.

A. 164,000 pounds in 1930 and 194,000 pounds in 1931.

By Mr. Smith:

Q. During those years from 1928 to 1932, of your own product which you made according to the two-liquid process, can you state how much of that was actually sold and how much of it was distributed in the form of samples?

A. I should say that during the first year, 1928, undoubtedly by far the greater part of the material we turned out was submitted in the form of samples. But when it came to 1929 and the 20,000 pounds, that was delivered in much larger lots and was done on a billing basis, as I recollect it.

Q. What about the other years when you used the two-liquid process? Was that sent out on a billed basis?

A. 1930 and 1931?

Q. Yes.

A. It was certainly sold in a direct way.

[fol. 58] Q. You have mentioned that a premium was charged for this product: do you recollect what that premium was?

A. It was in the neighborhood of three-quarters of a cent to one cent a pound, I think it carried.

Q. Approximately what was the capacity of the semi-pilot plant that was used for making these pellets under the two-liquid process, do you know?

A. I do not know.

Q. Do you know where that plant was located?

A. Yes, sir.

Q. Will you please state where it was?

A. At Easton, Pa.

Q. As a result of your sales of the pellet made by the two-liquid process did you find the public would not pay this substantial amount of increase in price for the black?

A. We certainly found a very great resistance to the idea of paying it, yes, sir.

Q. Do you have any record, Mr. Kitchel, where those pellets were sold?

A. I am sure there are records available but I do not have them with me.

Q. Do you know in a general way whether it was to the rubber companies or to the ink makers or just where they went?

A. I am sure it was most largely the rubber companies although I know some samples were submitted to manufacturers in other lines of work besides rubber.

Q. At the present time, Mr. Kitchel, is your company making or using the micronex beads sold for use in lacquer or inks?

A. They are not.

Q. Do you know of any reason why they are not sold in those fields?

A. We sell bead form of other grades to those manufacturers.

Q. Without designating the micronex beads as a trade designation or a particular type of black as well as the form?

A. It is any form, compressed, uncompressed or beaded. [fol. 59] Q. I have gained the impression that micronex beads referred to that in the pellet form. You have other products sold in the pellet form, is that correct?

A. That is correct.

Q. Would you be able to designate those by their trade names?

A. There is a variety. There are Excelsior beads which are sold to the paint trade. There is another form of very high quality that goes into the lacquer and protective coating trade.

Q. So far as you know, did you ever know of the bead product which is sold to the ink trade?

A. I believe we have furnished bead black to ink manufacturers for their use but that requires a certain specific manner of handling and formulating both. It cannot be done in the regular way.

Q. Does your company employ different processes for the use of these beads?

A. My company does not produce the beads.

Q. The company that produces the beads is what company?

A. The Columbian Carbon Company.

Q. In connection with the activities of the Columbian Carbon Company, do you know of any process other than this single liquid process which you have previously described as being used by that company in the production of this bead form of carbon black? • • • [117-121].

• • • A. I understand that they are working constantly with all kinds of different processes, methods and procedures. I would not know to what extent they may have applied those things.

Q. To your knowledge are any processes other than the single liquid process used in other products?

A. I should certainly so include any production of micronex beads for rubber work. The greatest part is by the single liquid.

Q. Is there any use of the two-liquid process of Wiegand-Venuto in connection with the production of those beads?

A. I would have no way of knowing that.

[fol. 60] Q. I assume that your company and the Columbian Carbon Company, in company with most other businesses these days, has some competition in the dustless form of carbon black, is that correct?

A. It is a sad truth, sir.

Q. In order that the record may be complete, I wonder if you would give us the names of other companies selling in competition with your company the dustless form of carbon black?

A. The United Carbon Company, the Huber Company, and I believe the Continental Company also offer it.

Q. Do you think of any other?

A. I do not recollect any right now.

Q. Have you had occasion to examine the products of these competitive companies visually in the way in which you would examine the beads made by the single liquid and the two-liquid process?

A. I have.

Q. How do these products compare with the products of the Wiegand-Venuto two-liquid process, that is, the first of your pellets? Would you say they are obviously similar or obviously different?

A. They are very similar except possibly as to the matter of the size of the pellets. As I recollect, the first two-liquid pellets we turned out were made out according to a certain general size. Later it proved that somewhat smaller ones would be more useable. It would be only a matter of difference in size.

Q. In connection with those four characteristics of the Wiegand-Venuto two-liquid pellet which you described, would you say that all these competitive products were or were not this pure carbon black that you mentioned?

A. As far as I know they were all pure.

Q. What about this proposition of being hard enough to stand shipping and handling?

A. Some of them appear to be hard enough and others do not appear to be hard enough.

Q. Do you at the present time have any recollection as to which are which on that?

[fol. 61] A. I just remember these various samples at different times in which some of them appear to have broken down to an extent during handling or shipment so there was some evidence of dust.

Q. You cannot associate that with any of your competitors' products?

A. No.

Q. What about the characteristics of the softness of the pellet so as to be readily dispersed?

A. They all appear to be soft enough.

Q. What about the spherical shape of the products?

A. There appears to be more variance than anything else. Some were smoother and some were harder but all seem to be free flowing and at least reasonably dustless.

Q. By "free flowing" you mean what you referred to in your direct examination as the flowability of products?

A. That is right.

Q. In connection with your sales and distribution of the Wiegand-Venuto pellets made by that two-liquid process back in 1928 and 1929, did you ever have occasion to examine those pellets in the plants or the laboratories of any of your customers after they had been shipped to them?

A. Oh, yes, it was a necessary part of the procedure.

Q. Did you observe any other appearances in them as the result of the shipments to the customers?

A. Not to my recollection. They seemed to be hard enough to have stood the delivery and just what we wanted them to do.

Q. They were still soft enough to be readily dispersed in their product?

A. Yes.

Q. Did you ever put the products through a screening test or anything of that nature to determine whether any of the pellets had broken up during shipment?

A. I did not, no.

Q. Did you ever see the results of any such tests?

A. I understand that such tests were run.

[fol. 62] Q. Was there any complaint from these customers as to the breaking up of the pellets during shipment?

A. I recollect none.

Q. How were these pellets supplied to the customers dur-

ing that period from 1928 to 1931, when you were operating on a two-liquid process?

A. In paper bags.

Q. There were no tank car shipments in those days?

A. There was not enough to fill a tank car.

Q. Do you know who was the first to suggest the shipment of these carbon black pellets in tank cars, what company?

A. I should assume that we were.

Q. About when if you know, was the first shipment made in tank cars by your company?

A. I do not recollect off hand. I would have to obtain that information; I do not have it with me in my records.

Q. I wish you would look that up if you can and if it is agreeable, to furnish that to counsel. We would like to have that information. Do you know who suggested the handling of the carbon black in the pellet form in its bulk handling system,—what company?

A. That was a matter that was discussed by ourselves in our own company in the very early stages after we found the flowability. I would not know how to determine that.

Q. Could you fix that with relation to whether it was during the time you were selling the beads of the two-liquid or single liquid?

A. They were certainly going on at the time of both,—the two-liquid and the single liquid.

Q. Did you advocate to the customers by furnishing these samples of the Wiegand-Venuto two-liquid process pellets that they install bulk handling equipment to facilitate the handling of those pellets?

A. We did not get to that point for the reason I outlined in my testimony.

Q. During your direct examination you testified to a booklet entitled "Flow Carbon." Do you know who wrote that?

A. That is a combination of authors. There were con- [fol. 63] tributions made by several. There are different headings in the book and different authors.

Q. You have read the book?

A. Yes • • • [122-127].

• • • Q. Can you tell me when the Columbian Carbon Company discontinued the use of the two-liquid process?

A. They started on the single liquid very early in 1932, so it must have been continued at the end of 1931, or was discontinued at the very beginning of 1932 or end of 1931.

Q. Do you know where the manufacture of pellets by the single liquid process was begun?

A. The single?

Q. The single liquid, yes.

A. I am not certain as to whether that single liquid material was produced both in Louisiana and Texas or only in Texas.

Q. Can you tell where it might have been if it was in Louisiana?

A. Undoubtedly around in the Monroe District.

Q. If it was in Texas can you state where it would be?

A. Probably in the Pampa District.

Q. Do you know on what scale the single liquid process was worked early in 1932, I believe you said it was adopted? To what amount of pellets?

A. To as much as we could sell.

Q. According to the notes which I took on your direct examination, in 1932 that was 281,000 pounds, is that correct?

A. Correct.

Q. What has been your relation to the Columbian Carbon Company during this period of time that you have been testifying about, Mr. Kitchel, particularly with regard to this period from 1928 through 1939 on which you gave us the figures of production?

A. The relation of agent to principal.

Q. Have you during that period of time been a director of the Columbian?

A. Yes, sir.

[fol. 64] Q. Is there any stock relationship between the Columbian Carbon Company and Binney & Smith?

A. Columbian has no interest whatsoever in Binney & Smith Company.

Q. Does Binney & Smith have any interest in the Columbian?

A. They own stock in that company. It is a listed stock on the Exchange.

Q. Are the activities of the Columbian in any way controlled by Binney & Smith?

A. No, sir.

Q. During the time that the Columbian Carbon Company operated under the two-liquid process of this Wiegand-Venuto patent did they pay a royalty to Binney & Smith?

A. We were very glad indeed to have them put that into factory operation so no license was charged.

Q. So then I understand that no royalty was paid by the Columbian Carbon Company to Binney & Smith for the operation from 1928 to 1931?

A. That is right.

Q. Has there been any royalty paid since that time?

A. No. We were most anxious to have them give us the product and put it up in that way and produce it for us to sell.

By the Court:

Q. Do Binney & Smith sell all of it?

A. That is right, sir.

Mr. Dike: We will agree it was all supplied by Binney & Smith. I think it so states there.

Q. During November, 1930, do you know whether or not Binney & Smith was an advertiser in the India Rubber World?

A. I feel pretty sure so. It was an advertiser in that for many years prior to that.

Q. Have you continued to advertise in the India Rubber World?

A. We feel we must.

Q. So it would take in the issue of May 1, 1933?

A. There is no break in our advertising as far as I know. It is a recognized trade journal.

[fol. 65] Q. You feel sure all these excerpts in this booklet were taken at a time when Binney & Smith was advertising in the India Rubber World?

A. I would be very much surprised if that were not so, yes.

Q. Do you contend that any black in the form of dustless pellets which were hard enough to stand shipment and soft enough to break down under slight pressure satisfied the demands of this Wiegand-Venuto patent? • • • [127-131].

• • • A. Material in free flowing dustless form that had the two properties of being hard enough and being soft

enough to fill the requirement. I would say there is one thing lacking,—is it pure?

Q. We will put the "pure" on it.

A. If it is pure carbon and if it is hard enough and soft enough, and has that flowability and is relatively free from dust, I would say that it is.

Q. Do you find those properties of the dustless product of the Huber Company or the Cabot Company?

A. In general I see no difference * * * [131-132].

* * * Q. Do you have any figures which would indicate the amount which was expended for advertising the pellet form of the carbon black during the years of 1928 through 1931?

A. I have no figures with me which would indicate that. I could only give a rough idea of what our advertising appropriation for rubber black at that time was.

Q. That would not show anything with reference to the relative amount to the compressed form or bead form,—those figures?

A. I would have to inquire.

Q. Will you kindly make such inquiry then?

A. That is, the amount spent in advertising on the pellet product?

Q. Yes, if you will get that for each year from 1928 through 1940, please. Are you familiar with what was done by the Binney & Smith firm on sales promotion on this pellet form of carbon black from 1928 to 1939?

A. Oh, yes, I am familiar with that.

[fol. 66] Q. Would you outline to us briefly what those sales promotion efforts were?

A. All kinds of advertising, direct mail, personal solicitation of accounts, conducting and demonstrating tests and everything of a conceivable nature that we could do to build up distribution.

Q. Have you any record as to how much was expended in these ways for promoting this sale of these beads of carbon black?

A. Not that I know of.

Q. Are such figures available?

A. I have no reason to think so.

Q. Will you make inquiry and let us know?

A. In other words, sales promotion expenses?

Q. That is right. I would assume from your outline of

the things that were done that was a rather expensive proceeding. Would that be a correct assumption?

A. We felt so at the time.

Q. It would be a rather substantial amount, I take it?

A. It seemed so, particularly during that period because that was just the bottom of the depression and everything seemed to be coming down around our ears and a penny then was of a pretty big size.

Q. In connection with these publications which you referred to in your direct testimony, particularly those articles in the India Rubber World on the dustless Micronex product, do you know whether the data for those articles was supplied by the Binney & Smith Company?

A. It was.

Q. Did you ever see the semi-pilot plant used in connection with the manufacture of these pellets by the two-liquid process?

A. Yes.

Q. Can you state whether or not the agitating equipment was similar to what you had used for demonstrating purposes?

A. I should say all sorts. I would not know which was the preferred one.

Q. Then, if I understand you correctly, the production of that semi-pilot product plant of the two-liquid process [fol. 67] used various types of agitators, mixers and what not to produce the pellets?

A. The process was in a continuing state of experimentation and everything was being tried all the time.

Q. Why was it necessary to do so much experimental work in that plant?

A. I suppose because they were after the very best possible results.

Q. Do you know whether or not there was any difference in the product which was offered to the trade under that two-liquid process as the result of these different conditions in the semi-pilot operation?

A. I do not know.

Q. In your direct examination you mentioned that the pellet product had a density of 27 or 28 pounds per cubic foot. I did not catch whether that was the product of the two-liquid operation or the old single liquid product.

A. I know of no difference between them. If there is I have not been acquainted with it.

Q. So the figure of 27 or 28 pounds per cubic foot you think would be either the product of the two-liquid or the single liquid?

A. As far as I know.

Q. Prior to the time that the black in pellet form was distributed to the rubber trade I believe you said that the compounding was carried out in separate rooms in some of the rubber companies, is that correct?

A. Some of them had separate rooms, yes.

Q. Do you recall about when you saw separate rooms in the rubber companies' compounding?

A. That was so long ago I would hate to hazard a guess on that.

Q. In connection with all these difficulties on which you elaborated in connection with the use of carbon black around the rubber mill, do you know of any instance where the rubber people did not use carbon black because of those reasons?

A. I would say it caused a serious delay in accepting the advantages that carbon black gave because of the nuisance. I know of no case where it prohibited its use eventually.

[fol. 68] Q. I believe you say you knew Geer, Knowlton and Hoffman?

A. I said I knew Geer.

Q. When did you first become acquainted with Mr. Geer?

A. In 1913 or 1914.

Q. I note that the application date of the patent was 1916, that is, his Patent 1,245,700, so it would be prior to the time that patent application was filed when you first met him?

A. Oh, yes.

Q. Did you ever discuss this patent with Mr. Geer at any time?

A. No, we discussed only the problem.

Q. Did you ever discuss the Knowlton and Hoffman patent with Mr. Geer?

A. No.

Q. In connection with the Wiegand-Venuto two-liquid process which was used in the pilot plant do you know whether any organic liquids other than gasoline were used in the commercial production?

A. Not to my knowledge.

Q. In the single liquid process which now produces Micronex beads, do you know of any organic liquid being used?

A. No.

Q. Do you know of any wetting agent or liquid other than water being used in that process?

A. No * * * [132-137].

* * * Q. In your direct testimony you referred to the fact that a license had been taken by the Thermatonic Carbon Company?

A. That is correct.

Q. Do you know when that license was taken?

A. Just recently.

Q. This year or last year?

A. This year.

Q. 1941?

A. Yes.

Q. Does that license cover any other patent than this Wiegand-Venuto?

A. It covers this Wiegand-Venuto and others.

[fol. 69] Redirect examination * * * [138].

* * * Q. The Thermatonic has recently gone into the business or is about to go into the business, and that is the reason for the license at this time?

A. I do not know of them making any pelleted black prior to this time. I understand they are now interested in getting into that field and that that is why they took the license.

Q. You were asked if you ever talked to Mr. Geer about the proposal detailed in his patent and you said you had not. Were you not thinking of the years you had been talking about?

A. Yes, 1915.

Q. In recent years you have talked to Mr. Geer about that?

A. Oh, yes.

Q. And if counsel wishes to inquire about it, you are ready to tell him what Mr. Geer has stated?

A. Oh, yes * * * [138-139].

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* * * Testimony of Webster Jones.

Direct examination * * * [141].

* * * Q. Please state your name.

A. Webster Jones.

Q. Your place of residence, Doctor.

A. Pittsburgh, Pa.

Q. What is your present occupation at Pittsburgh?

A. Dean of Engineering, Carnegie Institute of Technology.

Q. You graduated from a technical school.

A. Yes.

Q. From what school?

A. I graduated from the University of Missouri in 1908.

Q. What is the general nature of your work from the time of graduation up to 1918?

A. In 1908 and 1909 I was teaching at the University of Missouri. From there I taught at Purdue University. I undertook graduate work at Harvard University and I [fol. 70] taught at Harvard. I also taught at the University of Maine and the University of Montana.

Q. That covers the period until 1918?

A. Yes.

Q. What were you doing in 1918 and 1919?

A. In 1918 I entered the service of the United States as first lieutenant in the Chemical Warfare Service.

Q. When were you discharged from the service?

A. I was discharged on March 6, 1919.

Q. Now will you tell us what you did immediately following that?

A. On April 1, 1919, I entered the employ of the B. F. Goodrich Rubber Company.

Q. And continued in the employ of that company for what period?

A. Until 1932.

Q. Now will you state briefly in what capacity or capacities you served and in what branch of the Goodrich Company business over that period of years 1919 to 1932?

A. I was invited to join the Goodrich Company by Dr. W. C. Geer. I entered the research department and from the Research Department I became manager of the General Testing Laboratory and the raw materials division. From that place I was advanced to technical superintendent of the processing division. From that position I became general superintendent of the processing division. That position I held when I left the Goodrich Company. I left to become Dean of Engineering at the Carnegie Institute of Technology.

Q. Immediately following your entrance into the Goodrich organization in 1919, did you become acquainted with

the practice of the company in the use of carbon black in rubber manufacture?

A. I did, sir.

Q. What did you find that the Goodrich Company was using at that time, what form of carbon black?

A. At that time the Goodrich Company was passing from uncompressed carbon black to compressed carbon black.

By the Court:

Q. What time was that?

A. 1919, sir.

By Mr. Edmonds:

Q. Were they using carbon black in large quantities?

A. They were.

[fol. 71] Q. Will you tell us what were the circumstances incident to the use of carbon black as Goodrich used it?

A. Goodrich used uncompressed and compressed carbon black when I first entered the industry, and then the compressed black. The black is absolutely essential in obtaining the best quality in tire tread and it was used in very large amounts. In the beginning 20 to 25% of the black was used on the rubber. As the work progressed more and more carbon black was used. It was a gradual change in the years I was with the Goodrich Company. Ultimately as high as 45 to 50% of black was used on rubber. That is, 100 parts of rubber to 45 to 50 parts of black.

Q. Now will you tell us something about the condition that prevailed in the operation of the Goodrich plant where the carbon was introduced into the rubber, what sort of machines were used and what was done with the carbon black and anything incident to that use?

A. The various processes in the preparation, say of a tire, are to determine, first, the formula. That is done by technical men. After the formula is arrived at by the technical men through various methods used in the art the formula is sent to the compounding room. In the compounding room the materials are exactly weighed out and then they are sent to the mill room. In one of the mill rooms at the Goodrich Company, there was a large mill room, 44 mills with 84 inch rolls. Now these rolls turn in opposite directions. The rubber is placed on the roll and masticated,

that is, the rubber is gotten over the entire mill roll. Then after a certain amount of mastication the black is added. During the addition of this black it flies in every direction, as was witnessed here in uncompressed and compressed black yesterday. It is a terrible nuisance in the mill room and it does not stop at the mill room; it flies over the whole plant and settles wherever it might fall and contaminates people and materials throughout the plant.

After the black is mixed in on this 84 inch roll,—there are two rolls, as you saw yesterday, in a small mill, which was a 12 inch mill. The back roll is driven and the other [fol. 72] is not. You can adjust the distance between the rolls by screws on the side of the mill; you can adjust the distance between the two rolls. By just mixing the rubber and then pouring on the black, clouds of dust will form, and, when the black is pretty well taken up, the rubber is cut back and forth by a mill man. In that way it is rolled and it enables the black to be mixed into the rubber. Underneath the mill rolls is a pan called a mill pan which collects the part which is not at the time worked into the rubber. That is then put back on.

Q. When the rubber goes into that mill it is in a crude form?

A. Like the smoke sheet that you saw. There are two principal grades of crude rubber, the pale crepe and smoke sheet.

I happened to be on the receiving end of this carbon black. That is, as general superintendent of the processing division I received the complaints, and it so happened that executive officers were on the fifth floor in a separate building from this mill room I am speaking of, and I got complaints from the executives, "why don't you cut down this black nuisance?" Of course the mill men did not like it, and they did not care to work in a dusty atmosphere.

Q. Were you yourself in and around the mill room day in and day out?

A. I had 1500 men working for me and I had to be on the job 24 hours a day.

Q. Did you wear a cap to keep it out of your hair?

A. I did not have to do that; the mill men did.

Q. Was that common?

A. Yes. In addition to that, when they got through their day's work that could not be washed off without much

difficulty. As you get into this black it is very difficult to get it off and it keeps ahead of where you are washing. If you have the carbon black on your hands it will creep up around your neck.

Q. Did that condition continue right along from 1919?

A. Yes, sir.

Q. At any time from 1919 or at any time thereafter did Goodrich make any use of any form of carbon black [fol. 73] that had a binder mixed in with the carbon particles?

A. The rubber compounder uses his material straight. He does not want any binder in his materials. He will do his own compounding. Compounding is an exact science.

Q. As a matter of fact no such binder was ever used in the carbon black at Goodrich?

A. No, sir, not while I was there.

Q. Along in the '20's did you make some proposal with reference to ameliorating these conditions due to dustiness which Goodrich adopted?

A. The dustiness was a nuisance. It so happened that when I became general superintendent of the processing division, in addition to the suppliers being stirred up about this black nuisance I did something myself. I was responsible for the building of a separate mill room for mixing this black material. We spent a million dollars to just put this black off to one side.

Q. What was the basic thing in that expenditure?

A. The segregating of black to avoid the dust nuisance, the nuisance to the workmen and the nuisance to the executives, also the wear and tear that this dust would cause on the machinery, also discoloration.

Q. Was that found to be a menace to the health of those engaged?

A. Well, it was thought to be, yes.

Q. There was some doubt as to what effect it had as to inflammability and explodability?

A. We had the same sort of sign I see here on the wall, "No Smoking," and I thought it was right.

Q. You spoke of Dr. Geer. Did he continue in the Goodrich association for some years after you went there in 1919?

A. Yes. My impression is that Dr. Geer left there around 1925.

Q. Up to that time he had been in charge of research and development at Goodrich?

A. Dr. Geer was vice president in charge of research [fol. 74] and development, and he is generally given credit throughout the rubber industry for introducing research into the rubber industry.

Q. Was Hoffman there while you were?

A. Yes. Harry Hoffman is one of my close friends. We worked in the Goodrich organization at the same time and both of us reported to Dr. Geer.

Q. You say you left the Goodrich organization in 1932?

A. 1932.

Q. At that time or at any time before you left had anything come under your examination in the way of a carbon black that is associated in any way with this dust problem?

A. Yes, sir.

Q. Tell us about that.

A. In the Goodrich Company we turned out in the neighborhood of 20,000 tires a day. That is a large order. These operations must be conducted with safety. In other words, you do not jump into the use of a new compounding material,—it takes time. Roughly speaking, it takes on the average two years to make a major change in any compounding. In tires that is especially true because before you use a new formula it has to be road-tested. That is, they send out cars and the question is, "Will this material stand up in use?" There are a large number of technical men in Goodrich, perhaps 400 or more technical men, following the quality of raw materials, the quality of new products and the quality of finished products.

Q. Will you bring that down to this innovation that was connected in some way with the dust nuisance?

A. After I had spent this million dollars for the segregation of black, a new product was presented to the raw materials division, and it happened to be under my charge. Here was carbon black in the form of pellet. Investigations were in progress when I left the Goodrich Company as to the utility of this new form of carbon black, that was called dustless, in the form of pellets.

Q. How far had that investigation gotten by the time you left the company?

[fol. 75] A. It had not gone beyond the experimental stage. That is, it had not gotten into wide use.

Q. Then that was in 1932, and it was at that time you went to Carnegie, where you have since been?

A. That is right.

Q. During your years at Carnegie have you maintained your connection with the rubber industry?

A. It is a very fascinating industry. In order to maintain my contacts I have for the past several years put out the annual survey of the rubber industry, which keeps me in touch with the rubber men in the industry. They are very kind to me. They assist me in the preparation of this annual survey.

Q. In that connection do you go back to the rubber plants from time to time on visits?

A. I do.

Q. In your more recent investigation of rubber plants since 1932, on your return trips, so to speak, what can you tell us about the practice?

A. It would be a joy to work in a rubber plant where black is being mixed because I have a very distinct memory of the dusty condition and the raw hiding I received when I was head of the processing division at Goodrich. I have the entree to the rubber factories and it is a marvelous improvement.

Q. State just what the improvement is.

A. Freedom from dust, sir.

Q. What is that due to?

A. To the special form of the material that is now being used, which, it is claimed is its free flowness. In other words, we had to handle the compressed black in sacks and the sacks were cut and the material spilled on the mill. That is no longer necessary. This material can be automatically weighed. If it is automatically weighed the dust nuisance is avoided.

Q. Is that dustless effect due to the use of that carbon black which you had under examination in 1932?

A. Yes.

[fol. 76] Cross Examination • • • [141-151].

• • • Q. You have described in some detail, Dr. Jones, the action of the old type of roller mill that was used in the rubber plant?

A. Yes, sir.

Q. I wonder if you would describe for us the action of the modern Banbury mill?

A. Yes, sir. When I went with the Goodrich Company the Banbury mixer was not in use.

Q. That would be in 1919?

A. In 1919. The first Banbury that we used was obtained from a neighboring company, that is, a neighboring company in the experimental stage of this Banbury mixer, and they could not make a go of it.

Q. Can you fix the date of that approximately?

A. I would say in 1925, or maybe, 1927. When I took over the mill rooms we had two 27 Banburys in the mill room. When I put into effect this segregation of black we used Banbury mixers.

Q. That is, in place of the old type roller mill?

A. Wait a minute. That is a yes and no question. I will tell you how Banbury operates. The Banbury, instead of having cylindrical rolls, has rotors. The rotors are enclosed.

By the Court:

Q. What is the difference between a rotor and a roll?

A. The rolls do not touch.

By Mr. Edmonds:

Q. Is not this a picture you can use?

A. Yes, this is a rotor. The rotors go in opposite directions.

By the Court:

Q. The witness is referring to page 10 of "Banbury Mixers," Bulletin 171?

A. Yes, sir. Here is the hopper the material was put through. This is a ram. The ram is closed after the materials are added. All the materials are not added at once. You put in the rubber first and you must get the rubber in the proper condition so it will take up the pigment. These are rotors. They do not touch and they can be adjusted [fol. 77] with screws so you can get any bite you want. In that connection, let me say that the cooling of the stock,—there is heat generated when black is mixed into rubber and cold water is put through the mill rolls. That is a Banbury mixer. There are various sizes of Banbury mixers.

Mr. Smith: At this time we would like to offer, as "Defendant's Exhibit No. 1," Bulletin No. 171, "Banbury Mixers," page 10 of which was referred to by the witness in his previous answer.

(Defendant's Exhibit No. 1, filed.)

Q. Is there some other page you want to refer to?

A. On an 84 inch mill, 200 to 250 pounds of stock can be readily handled. In this Banbury mixer you can handle 750 pounds to 1,000 pounds. The time of mixing a tread stock on the roller mill is about 25 minutes. The time of mixing the black into the rubber in the Banbury mixer, 750 pounds say is 11 minutes. In conjunction with a Banbury mixer, you always find 84 inch mills because when a batch comes out of the Banbury it is in chunks and you cannot handle rubber in chunks so it is sheeted out on 84 inch mills. In connection with the Banbury mixer for the mixing of stocks, it does not eliminate the black nuisance.

Q. When you were at Goodrich you fixed the time for the first work with the Banbury at around 1925 to 1927?

A. Yes.

Q. When was it you put up this new mill?

A. In 1928.

Q. I asked you a question previously as to whether you used a Banbury mill or an old type open roller mill?

A. We used the Banbury in connection with 84 inch mills.

Q. That is the roller mill for sheeting out the rubber?

A. Yes. It was cut afterwards, and the cut is very essential in the mixing of stocks. In all pigments added to rubber you must have complete dispersion.

Q. So far as those 84 inch mills in the new mill room are concerned, were they ever used for compounding carbon [fol. 78] black with rubber?

A. Were they ever used? We used the Banbury mixer in conjunction with 84 inch mills.

Q. My question is directed to this one point, as to whether or not carbon black was compounded with rubber on what you term an 84 inch mill?

A. It could have been done.

Q. Was it done?

A. We would not leave the Banbury stand idle. The 84 inch mills—on that it was the same as in other parts of the factory. The rubber industry is a too-competitive

industry and you cannot let machinery lie idle. These Banbury mixers cost $60,000,—figure it out.

Q. How many Banbury mixers did you have in your new mill; Doctor?

A. Two.

Q. How many of these 84 inch roller mills would you have in that mill room?

A. The better the mixing the fewer complaints the processing division will have. The ratio is one Banbury to three 84 inch mills. This is the mixing end of it. The more you mix the more it costs you. If you mill a little you get in trouble with the product. If you lose a customer's acceptance you do not sell him.

Q. At the time you put in the mill room, what was the type of carbon black you were using in the mill room?

A. Compressed black.

Q. Is that what is referred to as the heavy compressed black. You can tell this from the sample, Plaintiff's Exhibit No. 9.

A. You are acquainted with the different kinds of black?

Q. Yes.

A. There is a black called uncompressed black that weighs 13 pounds to the cubic foot. The compressed black I am talking about weighs in the neighborhood of 25 pounds to the cubic foot, supplied by anyone of the suppliers of carbon black to the rubbeer industry.

Q. When you go in the mill room was there any change made in the type of compressed black that you used in the [fol. 79] mill room over what had been previously used?

A. In the open mills.

Q. Referring to this diagram on page 10 of the Banbury bulletin, Defendant's Exhibit No. 1, I note reference to what is termed an air-operated ram. I did not get very clearly the operation of that ram. I wonder if you would please describe it to us.

A. All right. First the rubber is added to the Banbury mixer.

Q. Is that added to what is termed the hopper?

A. Yes, the rubber must be masticated before you add pigments. That ram serves a double function. That is, it puts pressure on the stock at the proper time. That is one. It closes the mixer and it goes down on there. When it goes down, "Whew," a cloud of dust. I spent $30,000 to put in the Pangborn dust collector.

Q. Would the dust collector have a connection with the Banbury mill similar to that which is on this diagram with the fan to reduce dust?

A. Yes. The operator works the ram. There are specific directions on the mill, the rubber on the 84 inch mill. That is a matter of testing. It is followed by the technical men throughout the whole rubber industry.

Q. When the carbon black is added to the rubber in the Banbury mill is that also added to what is termed the feed hopper?

A. Yes, sir.

Q. After it is added through the feed hopper and the ram is closed, is there any further difference that is noticeable?

A. Not after it is mixed in.

Q. How long does that take after the ram is closed and the mill is running?

A. The cycle is approximately 11 minutes. You could run it 15; you could run it as long as you wanted to. The longer you run this expensive mill operation the more it costs you, so you have technical men that decide when it is enough.

Q. So far as the Banbury mixer is concerned, was there any difficulty in breaking up chunks of carbon black that [fol. 80] may have been wetted with water or that had a high moisture content?

A. No compounder of my acquaintance would add water to a black. These materials follow specifications. The raw material division buys black, if that is what you are talking about, under specifications.

The Court: Do you mean a black which has been constantly wet or has been wetted by some process?

Mr. Smith: Either way. There were instances of rejection of black because of accidental wetting and then there are also instances of intentional wetting.

The Court: The question is whether the Banbury machines would crush.

A. Judge, we would reject that and not use it. We would not get that far. I would not be that dumb.

Q. Is there any difficulty crushing these micronex beads of the plaintiff in the Banbury mills of the plaintiff, if you know? * * * [151-158].

The Court: • • • I will permit the question, is the Banbury an effective function in breaking up the micronex beads?

A. Yes, sir.

By the Court:

Q. Is it a fair statement to say that in the use of what you might term "dusty black," which includes the compressed form which was used when you left, that the Banbury machine ameliorated the dust nuisance but did not cure it?

A. Yes, sir • • • [159].

• • • Mr. Edmonds: We have an admission from the defendant that this is a sample of the defendant's product. I will offer this in evidence as "Plaintiff's Exhibit No. 20."

(Plaintiff's Exhibit No. 20, filed.) • • • [160].

• • • Testimony of Wm. B. Wiegand.

Direct Examination • • • [160].

Q. What is your name?
A. William Bryant Wiegand.
Q. And your residence?
A. Old Greenwich, Conn.
Q. Are you a technical graduate?
A. Yes, sir, University of Toronto, Canada.
[fol. 81] Q. What year?
A. 1912.
Q. And your present official capacity?
A. Director of Research, Columbian Carbon Company.
Q. You are the same William B. Wiegand who is one of the patentees in the patent in suit?
A. Yes, sir.
Q. I take it we may understand that you have given some years of study to carbon black, 20 years, more or less?
A. Yes.
Q. Will you look at this container of pelleted carbon black bearing the inscription "United Carbon Company; Beads, Exhibit No. 20"; have you examined this?
A. Yes.

Q. Will you state whether or not this carbon product is substantially pure carbon?

A. Yes, sir.

Q. Is it ppure carbon in the sense that it comes within the rather rigid specifications of the rubber companies which they apply to the carbon blacks they buy?

A. It does.

Q. Are these beads hard enough to stand the rigors of handling, manufacture and transportation to the users?

A. There seems to be no doubt about that.

Q. Are they soft enough so they will disperse readily and freely in rubber manufacture?

A. Yes, sir.

Q. It is quite obvious they are flowable?

A. Yes, sir.

Q. Are these pellets of a spongy or a porous nature in the interior?

A. They are.

Q. As to the pellets themselves, are they approximately 1/16 of an inch in diameter?

A. Yes, sir.

Q. What shape are these beads, Mr. Wiegand?

A. I suppose you would call them spheroidal. Roughly speaking they are a rough sphere and are nearer to that than anything else, I should say * * * [160-162].

* * * (Plaintiff's Exhibit No. 21, filed.)

[fol. 82] Cross-examination * * * [162].

* * * Q. You are one of the patentees in the patent in suit, are you, Dr. Wiegand?

A. Yes, sir.

Q. What examination have you made of these beads that were produced by the United Carbon Company?

A. You mean chemical or physical?

Q. The question is clear, can you answer it?

A. They are at least of rounded appearance in shape. We have examined them along the lines used by our customers in the way of establishing the requisite purity and we find that on that test they meet these and similar tests which the trade usually imposes, such as the amount of extractable matter, ash residue and tests of that kind. We did tests confirming that and it is almost self evident from

the density situation of pellets of this kind that they are porous or spongy.

Q. What test did you make to determine these were spongy or porous?

A. There are various ways of testing that and one means a great deal to me personally. It is just a straight consideration of the density. If carbon black of the density of 1.8 were solid, a cubic foot would weigh about 112 pounds. If the black were not solid but in pellet form, allowing for the air spaces between the pellets, assuming the pellets were solid it would weigh about 87 pounds to the cubic foot.

Q. What size pellets are you considering?

A. It does not make a great deal of difference.

Q. How much difference does it make between a sixteenth and 1/32?

A. Theoretically speaking, if they were all uniform you would never notice the difference. If you have a mixture of big ones and small ones, those figures would be susceptible of some correction. They would weigh 87 pounds, within one or two per cent. of it, but they actually weigh 26, 27 or 28 pounds, therefore they would not be more than one-third carbon. If they were pure carbon they would weigh about 87 pounds to the cubic foot; they weigh about [fol. 83] one-third of it. That is one indirect test of porosity. You can do a sucking up test and you can also actually displace the air by suitable wetting. I have done all those.

Q. What was the result of those tests?

A. They showed porosity.

Q. You are one of the patentees of the patent in suit, you stated?

A. Yes, sir.

Q. How does the flake form of black which you described in your patent differ from the pellet form and the various characteristics mentioned in Column No. 1, in which you did not mention them as coming from Column 1 * * *, [162-164].

* * * Q. Will you look at this specimen I hand you and tell me if that has the same characteristics as the sample you have just looked at, which was Plaintiff's Exhibit No. 20?

A. These are the beads that are stated to be standard United beads. May I inquire as to how far you want me

to comment upon that? I can only say that they have a general similarity.

By Mr. McLean:

Q. Would some light be helpful?

A. I can see all right. I do not want to make a snap judgment. I want to keep within what I can safely say. They have the appearance of pellets, they seem to flow. I would say offhand they have the same general appearance.

By Mr. Dike:

Q. If you assume they are pure carbon black they meet the other requirements that you have testified to in regard to Exhibit No. 20?

A. By no means. The other three questions,—as to flow I would say they show all the external symptoms of flowing. Whether they are hard enough or soft enough is another matter.

Q. Will you examine that and see?

A. I could not give you a final answer by examining that on the desk.

Q. What would be necessary to know?

A. I would have to know if they would stand transporta- [fol. 84] tion and the handling which is customary with carbon black. I would have to know whether in a wide series of rubber compounds, ranging from very hard ones to very soft ones, under those conditions they would disperse.

Q. Roughly, can you determine that?

A. We went through a great deal of that in that early stage of development. That was the greatest single remaining problem we had. When we got these pellets, the dust problem was solved, we felt. We took months and months to find out about softness; we sent bags to rubber companies on both sides of the Atlantic to see if they did have the softness and reinforcing qualities. They put them through the tire treads, which was the hardest type of compound, and in the shoe industry, representing soft compounds. Some people took years before they were convinced these pellets would disperse in the type of rubber they used. The softer the rubber the more difficult it is to take the pigment and get it distributed. The above tests are the tests which anyone would have to go through to

settle the question as to whether this product or any other product is what you would call "soft enough" to disperse.

Q. The product made by you is a dispersable product, is it not?

A. Yes, sir.

Q. That is one of the essentials of it, of course?

A. Yes, sir, most assuredly a prime essential, I would say.

Q. Can you tell by a microscopic examination whether a particular thing is spongy and porous?

A. I have never regarded a microscope as definitely a clue to that question. You can look at a pellet, you can see what it looks like and it is somewhat spongy but I have never found it quantitative but have found it rather treacherous. I lean to more quantitative things than a microscopic examination.

Q. How can you tell if a thing is spongy or porous?

A. I did not write the claims but the words to me mean the same thing.

[fol. 85] Q. You swore to them did you not?

A. Yes, sir.

Q. You understood what you swore to at that time?

A. I am telling you now what I understand. I take "sponginess" and "porosity" as meaning somewhat the same thing,—capable of absorbing, like a sponge.

Q. You do not refer to it then as an open or honeycombed structure or any thing of that kind?

A. No.

Q. You do not think you can tell that by microscopic examination?

A. Sometimes I think I can and sometimes not. It depends on how the sectioning is done. If you cut a pellet you get one thing, if you crush it you get somethnig else. If you put it into something that will open it out gradually like a mixture of turpentine and oil, or water, you get another condition.

Q. Tell us what those conditions are.

A. You can get the pellet to open up by degrees, whereas if you cut it it will fall apart.

Q. You do not think you can tell by breaking them up whether they are spongy or porous?

A. I would be afraid to say I relied on that method. It is a quick method. I prefer the more direct quantitative

ways by imbibition, by means of wetting it or something of that kind.

Q. Then you cannot tell whether a bead is spongy or porous definitely without some imbibition,—is that the right word?

A. Yes. I am afraid I will have to confess I am not able to make a complete diagnosis of a pellet by means of a microscope.

Q. You think that if you look at these beads under a microscope you could not tell if they were spongy or porous?

A. You mean as to whether they are more spongy or porous?

Q. No, whether they were spongy or porous within the meaning of your patent.

A. I prefer not to. I am not good enough with the microscope to commit myself on the basis of a microscopic analysis. I have not been able in twenty years to get from the microscope in the study of carbon black the help that other [fol. 86] people get in their subjects. The particles are beyond the range of a microscope. They are ten times smaller than a wave of light. If you relied too much upon a microscopic examination of a pigment you might be fooled.

Q. As I understood your direct testimony, you stated that Plaintiff's Exhibit 20 was spongy or porous: how did you determine that?

A. By these three methods which I have outlined. Knowing the density I may make a calculation of the amount of carbon that must be there and the amount of non-carbon spaces. Second, by means of sucking up to show that they were spongy. Thirdly, by actually seeing air issue from the particles when they were wet.

Q. When did you make those tests?

A. I made them on various occasions.

Q. And this sample here,—have you ever tested this particular sample? * * * [165-170].

* * * A. Yes, I examined the products which we are talking about, I believe.

Q. When did you make those tests?

A. I would say in the past two weeks.

Q. Will you produce the results of those tests, please?

A. I do not have the results here except to tell you what happened.

Q. Tell me exactly what happened. First, exactly what you did and what happened.

A. The density calculation I have already outlined. The imbibition test consists in taking——

By the Court:

Q. What is the meaning of it?

A. To drink up liquid. We take our own black for example,—that is one way of doing it. We take this particular product and allow liquid to suck into it.

By Mr. Dike:

Q. Describe your tests exactly and not in general. I want you to give me a definite description so I can go and make those tests.

A. We take a test tube and fill it to a certain level and take one product and then another product.

[fol. 87] Q. Yes, then the defendant's product?

A. Yes. As a matter of fact what we did originally was, we had a test tube filled with birdshot of the same pellet size, approximately, as these and fill to the same level in three test tubes and you pour a fixed amount of water in the three of them and check with the birdshot. If you have the water exactly right it will fill the little voids and rise in the form of a water level.

Q. A measured quantity of birdshot?

A. Yes.

Q. Then you measure the rise in the water?

A. Yes.

Q. Then tell me what you did.

A. You take carbon black of a particular pellet type.

Q. How much do you put in there?

A. The same amount and the same amount of water. You do not see any water, the water does not show. It is sucked up and it is imbibed by the little pellets, whereas in the case of nonporous materials like birdshot the water fills the voids. You do not get that level with the same amount. In addition to filling the voids between the pellets, this liquid is sucked up and you do not get any free liquid. That is the test I am talking about.

Q. You measure quantitatively the amount sucked up?

A. Yes. You can prove porosity in a very easy way. I imagine you can calculate the exact amount of porosity,

although I was not particularly interested in getting to the decimal point or exact amount.

Q. Did you calculate it at all?

A. No.

Q. Have you made any calculations at all?

A. No. I would say not in this particular method, the effect is so unmistakable.

Q. What is the next test?

A. The next test is to take preferably a low power microscope and in a little slide with a depression in it put a small amount of some applicable liquid such as linseed oil or turpentine, or even water and then drop a tiny amount of black into it and look at it. In a very few seconds you see gases exude from these pellets and come right out of them. In my judgment that is the most direct [fol. 88] test I have ever seen on this question of porosity.

Q. That is a comparatively simple test, is it not?

A. Oh, yes.

Q. What do you need for that test, Mr. Wiegand?

A. As I was saying, you need a microscope, any kind of a little low-power microscope that is convenient, a little slide with a hole, with a little recess in it, a supply of beads and a supply of some suitable liquid.

Q. What liquid?

A. I would suggest that we have some linseed oil, turpentine, alcohol and water.

Q. All of those?

A. I think they ought to be at hand, yes. Sometimes one works better than the other.

Q. And you would be willing to test the defendant's exhibit, I presume, and show us?

A. Yes, I would be willing, if necessary, to get those things together.

Q. Well, we have a microscope; but we don't have the liquids, but we can get them easily. Now, what degree of sponginess and porosity do you think is necessary to embody your invention—is necessary for your invention, I will say?

A. Enough to provide the first characteristic of a successful carbon black. Now, when you ask me to define the theoretical relationship between porosity and that all-important characteristic of disseminating through the rubber in such way that every particular individual particle of carbon black is lodged in its place in the rubber, which

in my judgment is the prerequisite of this reinforcement, sir, that everybody wants—if you ask me to define the relationship between that dispersability in rubber and porosity, I have got to admit my ignorance.

Q. So you don't know?

A. I don't know where the borderline is; no, sir. All I know is, if you have got—if you are within the proper range your pellet does a proper job in the rubber factory.

Q. Now, Mr. Wiegand, what about black which has been made into a paste by water, and then formed into pellets [fol. 89] directly?

A. You mean by the so-called single-liquid process?

Q. No, I am not referring to the single-liquid process. I am suggesting that you make some paste of carbon black and water and then convert that into a pellet and dry it. Would you consider that that is spongy and porous?

A. I would not know.

Q. You don't know?

A. I wouldn't know from the description you have given me what you would get.

Q. Do you mean to say in your study of carbon black of all these years you have never examined any pellets that were made from water and carbon black?

A. Oh, yes.

Q. First making them into a paste and rounding them up into pellets, and drying them?

A. I don't know exactly what you mean, for this reason: I don't recall ever having made pellets from carbon black and water, because a paste—well, it is a wet plastic mass, is it not?

Q. Yes.

A. Shiny. It has got enough water into it to make a paste. I couldn't answer your question.

Q. Well, let us say it is a continuous mass.

A. A continuous mass?

Q. Yes, for the sake of definition.

A. Yes. Now, if you ask me what the properties would be of any given paste when made into pellets, I would have to, in order to be accurate, to the best of my knowledge, say that I would have to see it, because it would depend on the type of process used, the type of agitation and apparatus used to convert it from the paste to the pellet.

Q. It might or might not be spongy and porous, you think?

The Court: You just answered that question. You said you didn't know.

A. Well, that is the point, your Honor. I don't. It might or might not; I suppose that is safe enough to say.

[fol. 90] By Mr. Dike:

Q. You measured the size of those pellets in Plaintiff's Exhibit No. 20?

A. No. If you mean by that, have I taken some of them and put them under a micrometer, to make a microscopic examination, I have not.

Q. To what extent have you measured them?

A. We do, as a routine thing, screen tests on carbon black. We find out how much there is on twenty, how much on forty, how much on sixty, and how much goes through at eighty mesh.

Q. Did you go through those tests on this Plaintiff's Exhibit No. 20?

A. I haven't in mind any particular screen test on this product, if that is what you are asking me. The question you asked me was whether they were approximately one-sixteenth of an inch?

Q. Yes.

A. Well, I am not going to swear they are not one-eighteenth of an inch, or a twentieth.

Q. Where do you think approximately one-sixteenth of an inch begins and ends?

A. I would say it depends on what you are talking about.

Q. Well, we are talking about carbon black pellets, of course.

A. We are talking about carbon black in pellet form?

Q. Yes.

A. Well, if I remember rightly, in our patent we said that we preferred them to be—I forget the exact figure—was it one-sixteenth, or something like that?

Q. Your claim states, "An article of manufacture, a pellet approximately one-sixteenth of an inch in diameter."

A. If I remember rightly, we said in our specification that we thought they should not be larger than a quarter of an inch; and that is my impression, that a pellet that approaches a quarter of an inch in size is not satisfactory any longer.

Q. You would say that did not conform within the term one-sixteenth of an inch, then?

A. That would be definitely outside the scope.

[fol. 91] Q. Definitely outside?

A. On the high side.

Q. Well, go to the low side. What is definitely out on the low side?

A. When you no longer have a condition of dustlessness.

Q. In other words, you can go down to what, roughly speaking, and give it to me in some measurement, so that I can have some idea of it.

A. As to what would fly?

Q. No, I want to know what you say is not one-sixteenth of an inch so far as this claim is concerned.

A. Well, as I said, I think if they get so small that they fly and they are no longer dust-free, then they are obviously not within the general idea of dustless carbon. I don't know where that borderline is, to tell you frankly.

Q. But roughly; you ought to know.

A. Oh, well, I would say——

Q. Would you say three hundred mesh?

A. Oh, no, I don't think you could get by with stuff that passed two hundred mesh even.

Q. You think it would begin to fly at two hundred mesh?

A. I think it would begin to fly at some point in between what these are and two hundred mesh; I don't know where that is exactly. It has not been one of our problems.

Q. In other words, you think the words one-sixteenth on an inch, as applied to that Exhibit No. 20, covers everything, say, from two hundred mesh up to a quarter of an inch?

A. I am a carbon black man, and I am afraid that I am disposed to look at these things in terms of whether they work or not.

Q. That is what I want your idea on.

A. Well, what is a mathematical equivalent of one-sixteenth of an inch—I don't know what to say.

Q. I am asking you as far as this carbon black is concerned.

A. Is it fair for me to say——

The Court: You can answer a very simple question. You testified that those beads were approximately one-sixteenth [fol. 92] of an inch in diameter. Now, I take it, you are

being asked within what tolerance, so to speak, do you refer; in other words, what do you mean by approximately one-sixteenth of an inch?

A. For a practical thing, or from a practical viewpoint——

The Court: No, you are not being asked about the function. You are just being asked what you meant by using that term in your testimony, from low to high, what do you mean by approximately one-sixteenth of an inch?

A. I am sorry; I had in mind the functions of the material, and I suppose something more definite than that should be said. I have already said it certainly ought not, in my judgment, to be bigger than a quarter of an inch, probably, to serve its purpose, and I would say to one one-hundredth of an inch.

By Mr. Dike:

Q. What mesh would that be, roughly?

A. Oh, I don't know; just to be frank, what the corresponding mesh is. There is a table.

Q. Yes, we have a table; but no matter about that now. Mr. Wiegand, can you give me some idea of where the dividing line is between porous and non-porous, as far as carbon black pellets are concerned?

A. I think I explained that we feel that porosity is the prerequisite to proper mixing or miscibility. If the product disseminates and mixes in the rubber and produces the reinforcing effects, for which carbon black is prized, my experience is that those pellets have the necessary structure.

Q. The only way to tell it is to try it then?

A. That is the only way to tell whether you have got the exact and necessary amount of internal looseness of structure; but if you want to demonstrate whether or not that porosity is there, you could do that without going through all that—without going to all that trouble by the bubble test of wetting, or by the imbibition.

Q. Now, you ordinarily refer to porosity, I take it, as a corollary of density, and you refer to it in terms of pounds per cubic foot of carbon black, don't you?

A. Yes, that is a rough idea of measure. The apparent [fol. 93] density of the whole mass, that is, but not the porosity of the individual pellet.

Q. I thought you told me it didn't make much difference about the size, and therefore that the question of weight per cubic foot would be the determining factor.

A. The weight? The amount of carbon black in a bag being in the general neighborhood of only one-third of what it would be if the black were solid, in my opinion is one of the most striking proofs of the presence of porosity inside it.

Q. Now, if black weighs 15 or 16 pounds to the cubic foot, pellet black, would you say those pellets were porous and spongy?

A. I would certainly expect them to be.

Q. And if they weighed 20 pounds, you would still expect them to be porous and spongy; would you not?

A. Most assuredly.

Q. And if they weighed 26 or 27 pounds per cubic foot, you would still expect them to be porous and spongy?

A. Yes.

Q. Now, if they weighed 35?

A. I would still think they were not all pure carbon inside them. I would still expect them to show positive porosity result.

Q. And what if they weigh 50 pounds to the cubic foot?

A. Well, I have never seen any—I am speculating on that now.

Q. Well, I want to find out what the limits are. I want you to give me your idea of what the limits are.

A. I have not seen any pellets that are denser than those which we are all accustomed to make in these days and I don't know about that.

Q. The real test is whether or not they will disperse in rubber?

A. That is the ultimate test, yes, for internal structure.

Q. Did you ever make a pellet that was not porous?

A. No.

[fol. 94] Q. Did you ever make one that was not spongy?

A. If you mean what I do by sponginess——

Q. I mean what you mean in your patent.

A. I mean imbibing, the property for which a sponge is ordinarily interpreted to mean in ordinary plain English.

Q. Well, if you could conceive of some pellets weighing 50 pounds to the cubic foot, would they imbibe water, do you think?

A. I don't know; I would be guessing.
Q. Well, I want you to guess.
A. If you want a guess, all right.
Q. That is what I want.
A. My guess is, they probably would.
Q. They probably would?
A. Yes.
Q. How about pellets weighing 75 pounds per cubic foot?
A. You are getting pretty high up in the realm of abstract theory there, now.
Q. I just want your idea.
A. Well, a pellet like that, I am afraid I am getting beyond my depth there. If you ask me to predict the properties of pellets weighing 75 pounds per cubic foot——
Q. Well, you would expect some porosity, would you not, Mr. Wiegand?
A. I prefer not to commit myself on that.

By the Court:

Q. By the way, what did you say a solid cubic foot of carbon black would weigh?
A. Well, in pellet form, all the same size, with no air spaces—I mean, allowing for air spaces, it would weigh about 87 pounds per cubic foot.

By Mr. Dike:

Q. About 87 pounds?
A. Yes.
Q. And that is where porosity or sponginess stops? You know it stops there.
A. If they are all perfect spheres, that is the theoretical figure. I have never known carbon black to consist of absolutely uniform spheres, to the extent that you are dealing with a commercial product, which consists of some bigger [fol. 95] ones and some smaller ones, and there is always a tiny component of dust. Those figures are necessarily approximations.
Q. But you did not answer my question. If you got to that point, those would have no porosity; isn't that right?
A. I just don't know, if you got up to 75 pounds, theoretically—well, I don't know, but theoretically you might think so.

Q. At the point—I forget the figure you mentioned——
A. Of solid carbon?
Q. Of solid carbon, then there would be no porosity.
A. That is right; I would not expect any porosity at about 87 pounds, on the assumption of uniform spheres.
Q. Now, porosity—when you refer to porosity, do you refer to absolute porosity, or do you refer to the ability to take up some particular liquid. I want you to define your terms, that is all * * * [170-186].
* * * A. Well, I don't quite understand what you mean by absolute porosity. I don't know what absolute porosity would be unless it is a pellet that consists of nothing but a hole * * * [186].

By Mr. Dike:

* * * Q. A pellet of a given structure has a given capacity for imbibing liquids, has it not?
A. I would think so, depending probably on the extent of its densification, yes.
Q. And the ability of the liquid to pass through the walls of the porous structure?
A. Well, that is what I broadly defined by "imbibing."
Q. Yes. Have you ever made a pellet which is not porous or spongy by your definition?
A. No, sir.
Q. So then that phrase, spongy and porous, defines anything from absolute density downward or upward, whichever way you might put it, being the dividing line of one side where a thing becomes a solid—do you understand what I mean?
A. Yes, and I hope I am not being too devious, but I [fol. 96] would like to reiterate that the expression, spongy or porous, as I understand it, means that the pellet has got sufficient softness to disseminate throughout the rubber and retain the all-important reinforcing properties of carbon black. Now, in trying to explain that property of sufficient softness, or mixing ability of carbon black in rubber, we have been talking about this question of porosity or sponginess. Now, the question of whether or not you can show that by other means, we have also discussed, and have illustrated it by means of imbibition and by means of pushing out little bubbles of air. Now, if you want me to commit myself to the extent of predicting within what range

the properties of internal structure, or porosity, in regard to its utility are, what we had in mind in this product, I certainly would not go over that range. I suspect that beyond a certain upper limit, there would be increasing difficulty in dispersion in rubber even, although that showed some imbibition, and showed some bubbles being pushed outward.

Q. About where is that limit; do you know?

A. I ought to know that, I admit, but I don't know.

Q. Have not you some idea? Was it about 30 pounds per cubic foot?

A. We have not, in our research program, tried to reach a pellet that would not work. We have been spending our time reaching one that would. I am, therefore, compelled to say that I don't know how far you can go to get a bad pellet from that angle.

Q. In other words, in your definition, you just gave me a minute ago, spongy or porous means that the pellet is capable of disintegrating and breaking up into its original or substantially into its original particles in the rubber mill?

A. I am going to try, if I may——

Q. Just answer my question. I think it is clear and I want you to answer that question yes or no.

A. I will try to answer that question in a way satisfactory to the Court and to——

Q. Now, just a minute. I want you to answer my question yes or no, and then you can give any explanation that you may think necessary * * * [187-189].

[fol. 97] * * * A. Yes. I will try to give you what you want on that answer. I would say that the expression, sponginess or porosity, has, in my judgment, two meanings: one is the purely chemical or physical meaning as to whether or not it is composed of solid carbon, or whether it has some non-carbon spaces in it. That, I would say, is the absolute or qualitative definition of porosity, subject to tests of imbibition and expression of gases. Now, the range of suitable porosity, in my judgment, would be one which would be determined by the results in mixing in rubber. That would fix the tolerable range, or the range of utility of that porosity.

Q. And that latter range is what is meant by the words, spongy or porous, in the claim of your patent?

A. It is what is in my mind as describing adequately that particular property of these beads; as to the claim wording, I don't profess to know.

Q. You are one of the patentees and you swore to this claim. I am asking you what you meant by the words spongy or porous.

Mr. McLean: Your Honor, the witness never swore to the claim.

Mr. Dike: All right; then we are out of court.

The Court: That may or may not be true; but he is responsible for what is in it. Answer the question.

A. That is my understanding of those words * * * [190-191].

* * * Q. That is rather ambiguous, and I wanted to make clear whether you meant the first half or the second half of that long answer you previously made; and, from hearing it read, I understand it to be the second half, but I wanted to be absolutely certain. Am I correct, Mr. Wiegand?

A. Yes * * * [191].

* * * Q. Were you and Mr. Venuto the first persons who conceived the idea of making a carbon black in pellet form, or were you the first to make pellets of a particular [fol. 98] character? I just want to get clear what you think you and Mr. Venuto were the first to do.

A. We knew of no pellets prior to those which we developed which had properties that we have been talking about.

Q. In other words, your answer is that you were the first to make pellets of the particular character of those claimed in your patent?

A. Yes.

Q. Did you know of pellets of carbon black, not of the character covered by your invention, previously? In other words, you did not know of any pellets of carbon black at the time?

A. No. Pardon me; what is a pellet? You have got to have that in mind a little bit in making an answer to that question. If you ask me under oath whether I have ever seen any carbon black in spherical form before, I have got to say yes.

Q. Yes?

A. I would not call them pellets, however.

Q. Well, what do you mean by a pellet?

A. Well, a pellet is the kind of thing that we make.

Q. But that is going around in a circle. I want to know what a pellet is.

A. What is the question?

Q. What is a pellet; what do you mean by a pellet?

A. I would call a pellet of carbon black a spheroidal shaped aggregate that had substance and strength to it.

Q. That is the way you used the word pellet in your patent?

A. Yes,

Q. Now, how much strength? How do you test strength?

A. Well, we have this riugh and ready test: does it survive under gentle rubbing of the fingers. I would not say that is an adequate test to predicate rubber behavior on, but it is a rough and ready test.

Q. And if it responds to that test, it is a pellet within the meaning of your patent?

A. I would be disposed to give it the name of a pellet if it did that, yes.

Q. If it goes to pieces——

A. If it goes to pieces, I don't think it deserves being called a pellet, just sort of a shapeless, structureless globule, [fol. 99] or mass • • • [192-194].

• • • Q. How do you think spongy and porous differ from each other, or what do you consider as to that? Do you consider them synonyms?

A. I consider that those words spongy or porous—well, spongy is porous, and a porous material is spongy in the sense that it is capable of soaking up things.

Q. Then the words are synonymous so far as you used them, to all intents and purposes?

A. Broadly speaking, I would think so.

Q. You don't make any fine distinction between the two?

A. No.

Q. Now, are the pellets made in accordance with your patent of uniform structure throughout, or is there a shell? You have studied them, and know?

A. Yes, we have done a good deal of poking around inside of our pellets and other pellets, and we find a lot of things in there if we are careful about it, in the way of opening them up by what we call exfoliation, where the pellet is put into a liquid and gradually allowed to unfold, we

see residual traces of a core and shell structure in nearly all of these commercial carbon black beads or pellets.

Q. I think you have said that Plaintiff's Exhibit No. 20 was of commercialiy uniform size. That is correct, is it not?

A. No; I don't know whether I did or not.

Q. Well, what is your answer to that question?

A. It is yes.

Q. Then Exhibit No. 20 is of commercially uniform size?

A. Yes.

Q. What variation in size do you think would take it outside that phrase, commercially uniform, as you use it in your claim?

A. Well, I am afraid there you have got me. I would have to consult the customer, I imagine, to see what he thought about it. There are none of them absolutely uniform.

[fol. 100] Q. Then the phrase commercially uniform in your claim does not mean much, does it?

A. It means a very great deal to us because it has got to be uniform enough to appeal to the customer.

Q. The only test, then, is customer appeal?

A. Satisfactory performance of the qualities for which it is bought and sold, flowability and things of that kind.

Q. What is the heaviest commercial pellet that you know, in pounds per cubic foot? A. I have no information on the relative—I don't know. I just don't know.

Q. How heavy a one have you seen; how heavy a black have you seen; that is, in pellet form?

A. I would have to consult the record on that; I don't know.

Q. What is your recollection?

A. I don't know.

Q. Give me a general idea. You can give me a general idea, can't you?

A. No.

Q. Is it 25 pounds?

A. No, I think it would be a lot heavier than that.

Q. How much?

A. 27 or 28.

Q. 27 or 28 is about the limit?

A. I think that would be the normal range.

Q. And why don't they make them heavier than that?

A. I am afraid that is a pretty hard question to ask me, and expect me to answer.

Q. I think you ought to know that.

A. Why don't we make them any heavier?

Q. Yes.

A. I thought you said "Why don't they."

Q. Well, probably I did; but I am sorry I misled you.

A. Well, I am very much afraid I have got to answer that, that nobody has ever asked for them. Nobody has ever asked me to make them heavier than that.

Q. Will you look at this specimen which I have here and tell me if you think that is commercially uniform, as the phrase is used in your patent? Take out some of it on a [fol. 101] piece of paper, if you want to.

A. You want to know whether it is commercially uniform, as regards size; is that it?

Q. Yes.

A. There are some big ones in here. My inclination, after examining that sample, would be to say that although it might handle all right in the operation, that there are too many big ones. I would be disposed, if we made them, to criticize the plant on the particular day, because I would say there were too many large particles in this particular lot.

Q. Then, you cannot say they are not commercially uniform?

A. Well, no, I would not go that far.

Q. So you think they are commercially uniform, then?

A. No, sir. They would not come up to my standards, as a technical man. I don't know what they would do in a plant of a customer.

Q. What do you mean by commercially uniform? You used the words in your claim, and I want to know what you mean?

A. By commercially uniform, I would say uniform enough to meet the requirements of commerce, in the sense that they are acceptable to our customers.

Q. And to find out whether they are commercially uniform, you have got to *to* ship them to your customers and find out whether or not they will use them; is that right?

A. I think that has got to be the ultimate and absolute test; yes, sir.

Mr. Dike: I will have the specimen shown to the witness marked for identification as Defendant's Exhibit No. 3.

(Defendant's Exhibit No. 3, filed.)

Q. You talked about dispersability in rubber. Is the dispersability in rubber you talked about a reduction of the carbon black pellet to original fine state of subdivision of the non-pelleted carbon black, or substantially so?

A. That is our theoretical view. The practical view is, does it give the road wear in tires? The test, the acid [fol. 102] test, is, do the tires wear as well on the road? Now, when you have said that, that is the starting point in our industry. Now, we explain satisfactory performance on the road on the theory that the particles are absolutely and uniformly dispersed; but there is no human being that has ever seen a carbon black particle dispersed in rubber that I know of.

Q. Now, does dispersability in rubber differ from dispersability in ink?

A. Broadly they are both the same; they both involve the same proposition. I presume you are speaking of pelleted carbon black?

Q. Pelleted carbon black, yes.

A. They both involve the question as to whether the pellets, on being subjected to normal milling procedure in either industry, perform in a manner similar to the older form of fluffy carbon.

Q. In other words, you cannot tell until you go and try it? A. I agree with that.

Q. You would define dispersability as being sufficiently fragile to permit reduction to the original fine state of subdivision upon the application of light pressure, would you not? * * * [194-200].

* * * A. No. Dispersability means the ability, when passed through the regular machinery used in the particular industry, whether it be rubber, ink or paint, to produce the effects known to be possible with ordinary fluffy black.

Q. Does that carbon black which is in Exhibit 20, have sufficient hardness to withstand shipment or handling without dusting? * * * [200-201].

* * * A. The fact that millions of pounds go through normal rubber bulk and flow handling equipment, go through

loading, transport in hopper cars and unloading in a manner satisfactory to the rubber trade, makes it impossible for my answer to be anything other than yes.

Q. Now, it is also, is it not, sufficiently fragile to permit reduction to its original fine state of subdivision on the [fol. 103] application of light pressure?

A. I have no direct evidence to give you on this particular sample, as to whether it fulfills all of these dispersive qualities which we have been talking about in the last few minutes, or not. The fact that it is used, to the best of my information, millions of pounds of it used to make tires, would lead me to suppose that the answer is yes.

Q. Then your answer is yes?

A. Oh, no, my answer is not that. My answer is that this material disperses under pressure of the hand.

Q. Will you look at the specimen I hand you and tell me if you consider the pellets in that to be commercially uniform, within the meaning of the words of your claim.

A. Those pellets are such giants in comparison with those with which I am acquainted, and which are ordinarily used, that I am afraid I would be a bit at a loss to say whether they are commercially uniform giants or not. Allowing for the difference in the whole size of them, I should think they are just as deserving of the term commercially uniform as ordinary pellets.

Q. Do they have a spongy and porous interior?

A. I would not be able to answer that, sir.

Q. Could you tell me by examination?

A. I can if I had time to go into the proper series of experiments, yes.

Q. Look at it and see what you think.

A. I don't have any equipment here to make any test.

Q. Do you want a microscope?

A. No; I have one here, but this is only a two-power lens.

Q. Could you tell under a microscope?

A. No.

Mr. McLean: The witness has already explained at great length that the microscope is not helpful to him.

The Witness: It would be unfair for me to diagnose a strange pellet in a snap judgment manner like that, and I would hesitate to do it.

Q. In other words, you could not tell whether those are spongy and porous or not?

A. Not like that.

[fol. 104] Q. Not like that?

A. No.

Q. And I suppose that would be perfectly true if I should show you a dozen other types of pellets; that you would say that you could not tell me by looking at them whether they were spongy and porous?

A. I am afraid I have got to answer yes.

Q. And you could not even tell by cracking them, could you, by cracking them open and looking at them under a microscope?

A. There are two aspects to this question of spongy, porous structure.

Q. Yes.

A. One is the absolute physio-chemical sense.

Q. Yes.

A. The other is the practical sense of internal structure, as reflected in performance in rubber. I could not do that right here.

Q. And your test is the performance in rubber, I take it?

A. I have explained all that.

Q. No, I don't think so.

A. I don't mind repeating it.

Q. Will you then, please?

The Court: No, I object to it. I think he has gone very thoroughly into the question of porosity and sponginess.

Mr. Dike: I take it his answer is yes.

The Court: His answer is he would not undertake, by casual inspection on the witness stand, to say whether these samples are spongy and porous, or not.

Mr. Dike: I will ask that the bottle shown the witness be marked for identification, Defendant's Exhibit No. 4.

(Defendant's Exhibit No. 4, filed.)

Q. When and where were commercial pellets first manufactured under your patented process?

A. Laboratory work in New York and pilot plant work in Easton, Pennsylvania; commercial production both in Easton and in Louisiana——

Q. Where in Louisiana?

A. In the Monroe District.

[fol. 105] Q. That doesn't mean anything to me. Give me the name of some town nearby.

A. It is one of those little towns within a ten-mile radius of Monroe; I don't remember the particular name of the place; just a carbon black plant. You know they are very little places, and I don't remember which one. I could look it up.

Q. Now, the other question was when. You did not answer that.

A. When and where they were first commercially made?

Q. Yes; at each of these places you have given, state when please.

A. Oh, pellets were made in 1927 in the laboratory and—well, I would have to consult my records; I don't want to make any mistake about this.

Q. Will you look it up and tell me when they were first made, when you first began to make them and sell them?

A. Yes, they were made in Easton very shortly after—I think in 1928 we shifted them from the laboratory to the pilot plant.

Q. In 1928?

A. In 1928.

Q. What was the capacity of that plant when you shifted it from the laboratory?

A. That was only a pilot plant. I would have to rather approximate it, but my estimate would be in the neighborhood of 100 pounds per day * * * [201-206].

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* * * Defendant's Witnesses

Testimony of Hilding Hanson * * * [208]

* * * Direct examination * * * [208]

* * * Q. Please state your name, residence and occupation?

A. Hilding Hanson; 801 Myrtle Road, Charleston, West Virginia.

Q. And what is your occupation, please?

A. I am now chief engineer for the United Carbon Company, of the carbon black division.

[fol. 106] Q. How long have you been employed by the United Carbon Company as chief engineer of the carbon black division?

A. I would say about—since about 1934.

Q. Prior to 1934, by whom were you employed?

A. I was still employed by the United Carbon Company.

Q. In what capacity?

A. As engineer.

Q. And when did that employment begin?

A. In August, 1927.

Q. Will you please outline briefly your duties as engineer for the United Carbon Company, from the period of 1927 until you became chief engineer?

A. In 1927 I started to work for the United Carbon Company at Borger, Texas, and there I worked with the construction superintendent, who was at that time constructing a carbon plant there, and I assisted him with any engineering matter that came up, designing certain details of the carbon plant, making drawings. Also I had charge of all of the electrical installation work, and also assisted with the surveying work.

Q. In connection with your duties as chief engineer of the carbon black department of the United Carbon Company, will you please outline briefly your duties?

A. Briefly, they are to supervise the operation of plants, supervise the designing and construction of these plants.

Q. Does that work include the designing of plants other than plants for the manufacture of carbon black?

A. Yes; also for processing carbon black.

Q. Will you please elaborate on what you mean by plants for processing carbon black?

A. By that I mean plants for processing ordinary channel carbon black into carbon black of the so-called dustless form.

Q. So that in your capacity of chief engineer, you are familiar with the manufacture of this so-called dustless carbon black by the defendant, United Carbon Company, Inc., are you?

A. Yes, I am.

Q. Now, will you please describe briefly the various processes which are used by the United Carbon Company, [fol. 107] Inc., for the manufacture of this so-called dustless carbon black?

A. Briefly, they can be segregated into two groups, the dry process and the wet process. The dry process is what is commonly known as the Cabot Process, and the wet process, there again you have two different processes, each one developed by us, the United Carbon Company.

Q. Will you please describe those two different wet processes you have just mentioned?

A. The first one is the so-called extrusion process. In that process we have a standard commercial extruding machine manufactured by the Pellet Manufacturing Company at Los Angeles, California, and a standard type of screen dryer and in operation the ordinary agitated channel carbon black is conveyed to a standard conveying system and into this mixer which I previously mentioned. There the black is mixed or agitated in conjunction with ordinary water. This wet carbon black mass is conveyed from this mixer into the extrusion machine. The extrusion machine consists essentially of a horizontal cylindrical die approximately, I would say, about 20 to 24 inches in diameter and approximately five-eighths of an inch thick. Into this die are drilled several hundred fine holes, approximately one-eighth inch in diameter. The carbon black is wetted carbon black. The wetted carbon black as it falls on top of the die is forced or extruded through these small openings by four horizontal rolls revolving on top of the die, with a very small clearance; at times they touch the die.

This is a continuous process. The black is continuously conveyed into this mixer and also continuously conveyed from the mixer into the extrusion machine. As the wetted carbon black mass is forced in the die, it comes out in the form of long strings like spaghetti, which break up by their own weight. When I say spaghetti, of course, they are not hollow; they are solid.

By the Court:

Q. And approximately one-eighth inch in diameter, you say?

A. Yes, sir. We have standard dies now with openings one-eighth inch in diameter. However, we have experimented with larger and smaller openings. As the pellets [fol. 108] leave the extrusion machine, they are dropped,

you might say, to a chute, and to the top of the screen in the drier. Here the pellets are dried as the screen travels through the entire length of the drier, and at the opposite end to the feed end of the drier, the pellets are discharged on to a belt conveyor. This belt conveyor carries the pellets to a conveyor elevator, which lifts the black up into a storage tank. It can be lifted up into either a so-called bag storage tank or a bulk storage tank.

From these two tanks, of course the black is packed in the bags or in the case of bulk storage tanks it is drawn into the tank cars.

Q. When they leave this place where they are forced through the holes, are they in a spherical form? I mean, are they in a cylindrical shape, or what is the shape?

A. They are not spheroids, no.

Q. They are not?

A. Not exactly. They are cylinders, I would say. They are like a pencil, about one-eighth inch in diameter, coming through the die. After coming through the die, and going through the chute on to the screen they break up into small short cylinders, the length of the cylinders being approximately the same as the diameter of the cylinder, so when they fall on the screen they are really a short cylinder one-eighth of an inch in diameter, and one-eighth of an inch long.

Q. What do you call that process?

A. The extrusion process.

Mr. Smith: I would like to have this marked for identification, Defendant's Exhibit No. 5.

Q. I hand you a container, which has just been marked for purposes of identification, Defendant's Exhibit No. 5, and ask you if you will look at that and tell us what it is.

A. I would say that is the dustless black produced by the extrusion process, after being dried.

(Defendant's Exhibit No. 5, filed.)

[fol. 109] Now, will you outline briefly the other wet process that you stated is employed by the United Carbon Company?

A. The other wet process, the processing or equipment consists essentially of two horizontal cylinders, one mounted above the other. Each cylinder is approximately 48 inches

in diameter, and twelve feet long. These cylinders are mounted on rollers, which turn the cylinders. In each cylinder are fastened round pins, which are approximately five-eighths of an inch in diameter, and 11 or 12 inches long. These pins are mounted radially inside the cylinders, fastened, of course, to the cylinder drum, extending inward. There are a number of those pins. I don't recall the exact number, but they are spaced about 3 inches apart, axially, and the periphery of the cylinder—well, there are twenty spaces; in other words, there are twenty pins in each roll.

By the Court:

Q. Are you describing the Sayre process?
A. Yes.

By Mr. Smith:

Q. Is that process you have just described used in other plants than the Sayre plant?

A. Yes, that process is also used in the McIlroy and two other plants.

Q. If you will refer to the chart which has been placed on the easel, I will ask you to explain what is shown on that chart.

A. Well, here are the two cylinders. This is the first cylinder and this is the second cylinder. Now, of course they are shown diagrammatically, and in reality one cylinder is mounted diagonally above the other. Here is shown the pins I spoke of, extending radially inward from the cylinder.

Here is shown the two rollers. Of course, there are two rollers on each side, on which the cylinders rest and revolve. Through each cylinder is mounted a shaft, and in which shaft is also mounted pins extending inward radially. The pins on the shaft are mounted in such way that they form a single spiral or tread around the shaft. The shaft in the upper drum is fastened in bearings outside the drum so that the shaft can revolve. In the lower drum the shaft [fol. 110] is mounted stationary, slightly offset to the center of the drum.

In operation the top drum revolves—it can revolve in either direction, but in operation we have arranged the top drum to revolve clock-wise and the shaft anti-clock-

wise, and in the lower drum it also rotates and the rotation is immaterial.

Q. You mean the direction of rotation?

A. The direction of rotation is immaterial. The top drum revolves approximately fifteen revolutions per minute. The lower drum approximately eight and one-half revolutions per minute. The top shaft revolves approximately twenty-two revolutions per minute.

At the end of one of the upper drums we have arranged a chute, which conveys the regular channel carbon black into the upper drum.

There is also a pipe extending to the opening of the feed end of the drum into the drum, in which we feed the water.

Q. Approximately how much black and how much water go into the upper drum?

A. The amount of water to black by weight is approximately 60 to 65 parts of water and 40 to 35 parts of black.

Q. How much black?

A. 40 to 35 parts.

Q. That is by weight?

A. Yes, by weight.

Q. Now, what sort of a mass is formed in that upper drum when you bring that amount of black and water together?

A. The rotation of the drum and the shaft, you might say, agitates and mixes at the same time the water and carbon black and in the beginning of the agitation and mixing operation the black and water has the appearance of, I would say, wet soil or loam, and as the black and water mixture proceeds along the inside of the drum and discharges from the drum you can observe fine pellets in that mass.

[fol. 111] Q. Now, is that mass what you would call a paste?

A. No, it isn't as wet as paste.

Q. What do you have in mind when the word "paste" is used?

A. I have in mind a mass that is more or less a plastic. I would say paste would have the consistency of butter, for instance.

Q. Is the carbon black and water in the upper drum a mass which has the consistency of butter?

A. No, it has not.

Q. Do you at any time ever have the mass in that upper drum to that consistency?

A. No.

Q. How is that controlled?

A. That is controlled by the flow of black and water through the upper drum. The flow of black is regulated by the speed of the conveyor conveying the black into the drum, and the flow of water is adjusted or regulated by valves in such manner that we have a constant flow of black and a constant flow of water.

Q. Now, what is the condition of that black and water mixture when it is discharged from the upper agitating drum?

A. As previously mentioned, it looks like moist soil or loam, and that is the whole mass. In that mass you can detect very fine pellets.

Q. Will you look at the container which is fastened to the chart directly beneath the words, "Upper Agitating Drum," and state whether or not that mass is the type of mass you just mentioned as coming from the upper drum?

A. Yes, that is the mass represented by—representing what I tried to describe as leaving the upper drum.

Q. When that mass leaves the upper drum what becomes of it?

A. It falls through a chute in the lower drum which is mounted directly below the upper drum. In other words, this drum is mounted below and it falls through the chute into this lower drum or cylinder.

[fol. 112] Q. Now, what happens in that lower drum cylinder?

A. This mass——

Q. That is the mass coming from the upper drum, now?

A. That is the mass coming from the upper drum, which is further agitated and broken up into smaller aggregates through the tumbling or rolling action of that drum and these small pellets that we found in the mass leaving the upper drum gradually increase in size and become larger and the mass breaks up into smaller agglomerates and also forms into pellets.

Q. Is there any additional water added to the lower drum, Mr. Hanson?

A. No, there is no water added to the black in the lower drum.

Q. Is there any black added to the mass in the lower drum?

A. No.

Q. Now, in carrying out that operation in the Sayre plant is any liquid other than water ever used for wetting the carbon black?

A. No, only water.

Q. Will you look at the little container underneath the words "Lower Agitating Drum" on the chart and state whether or not the material in that vial is typical of the material which is discharged from the lower agitating drum?

A. Yes, sir; that is typical of the material being discharged, yes, from the lower drum.

Q. What happens to that material that is discharged from the lower drum?

A. In addition to these two mixer drums we have a horizontal indirect heat type of drier. The drier is connected to the lower drum by a chute through which the pellets leaving the lower drum are conveyed into the drier.

Q. And what is the purpose of conveying the pellets to the drier?

A. The purpose is to bring the pellets into the drier to remove the water.

Q. Now, after the pellets pass through the drier what becomes of them?

A. As the pellets discharge from this cylindrical drier [fol. 113] they fall through a chute on a vibrating screen.

Q. And what is the purpose of the vibrating screen?

A. The vibrating screen is to remove those large lumps and agglomerates which have formed into pellets on our larger than 14 mesh.

Q. Is there any very large percentage of such large pellets or chunks which will not pass that 14 mesh screen?

A. There are approximately, in this plant, from 10 to 12 per cent. of coarse material that will not pass the 14 mesh screen.

Q. And what becomes of that material?

A. That material is conveyed back again into a smaller storage tank from which it is drawn and reprocessed through the whole system.

Q. Where does that black enter the upper drum? Is it in the portion marked "Loose Black" or some other place?

A. Where it is marked "Loose Black" it enters with the regular channel black.

Q. Will you look at the containers which are fastened on the chart, the one directly adjacent to the word "Screen" and state, if you can, what that specimen container represents, Mr. Hanson?

A. That material shown in that container represents the material as it leaves the drier but before it has passed through the screen.

Q. Now look at the other container which is also fastened to the chart, adjacent to the word "Storage," and state if you can what that specimen represents.

A. That material represents the product having passed through the 14 mesh screen.

Q. What becomes of that material that passes through the 14 mesh screen?

A. That material is conveyed over to a vertical elevator which elevates it up into a storage tank. This storage tank is a common storage tank for both bagging operation and tank car shipments * * *—[208-222].

[fol. 114] * * * Mr. Smith: I would like at this time to offer in evidence the chart which the witness just referred to in his testimony, as "Defendant's Exhibit No. 6."

(Defendant's Exhibit No. 6, filed.)

Q. I hand you a series of photographs which have been marked for identification as Defendant's Exhibits 7, 7a, 7b, 7c and 8. I will ask you to examine them and state if you can what they are.

A. The photograph Exhibit 7 shows, the top part of the photograph shows the lower part of the mixer bottom.

Q. Will you please identify that drum with reference to the chart Defendant's Exhibit No. 6?

A. The top part shows the part of the lower drum looking from there. The lower part of the photograph shows the inlet end of the rotary drier.

Q. Will you show that?

A. This shows one view of the rotary drier, the inlet end.

Q. Will you tell us what the photograph identified as No. 7-a is?

A. 7-a is practically the same view except it shows two bottom drums of the mixers; in other words, a pair of lower mixer drums.

Q. Will you please identify that particularly on the photograph?

A. The upper left-hand corner shows the rollers driving the lower left mixer drum, and the center part of the upper half of the picture shows the lower right-hand mixer drum with its driving rollers.

Q. What else is shown in the photograph?

A. The central part of the photograph shows the inlet end of the rotary drier.

Q. Will you please identify that on the chart Defendant's Exhibit No. 6?

A. The upper left-hand corner of the photograph shows that lower part of the lower mixing drum. Of course this is a diagrammatic view and can represent the lower mixer drums. The central part of the photograph shows the inlet end of the rotary drier.

[fol. 115] Q. Will you please refer to the photograph No. 7-b?

A. The photograph identified as 7-b shows the end views of the lower mixer drums as well as the rollers carrying the upper mixer drums. In the central part of the photograph in the foreground are shown the indicators by which we judge the amount of water going into the upper mixer drums.

Q. Are those indicators connected with the pipe which is labeled "Water" in the chart Defendant's Exhibit No. 6?

A. Yes, they are what is termed roto-meters and connected in series with the pipe feeding the water into the upper mixer drum.

Q. Will you refer to the photograph No. 7-c, please, show us which one that is and tell us about that.

A. The photograph 7-c shows the discharge end of the rotary drier at the left of the picture. In the center of the picture is shown the discharge tube from the drier. In the lower portion is shown the screen on which the pellets are screened after leaving the drier.

Q. Is that photograph 7-c shown diagrammatically on the chart Defendant's Exhibit No. 6?

A. Yes.

Q. Please indicate the portion.

A. The photograph shows the discharge end of the drier here and the screen which is shown there.

Q. Will you please take a pencil and mark on that photograph Defendant's Exhibit 7-c, the particular thing that you referred to as being the screen.

A. The screen is here.

Q. I believe that you testified that that screen was a 14 mesh screen?

A. It is 14 mesh. The screen that actually screens the powder, that is supporting a larger size heavy wire underneath.

Q. That is the actual grade——

A. The actual grade and is shown by the 14 mesh screen.

Q. Which is the screen you have marked?

A. Yes.

[fol. 116] Q. Will you refer to the photograph 7-d, please, and tell us about that?

A. In the foreground of the photograph 7-d is shown the tank cars in which the carbon black pellets are shipped from the plant to the consumer. Directly above and behind the tank car is shown a combination black storage and bulk system tank with its discharge spouts which are used for filling the tank cars. Directly behind the storage tank on the left-half of the figure is shown the building housing the drier, and behind that building is shown the building in which the mixer drums are located.

Q. Will you please indicate on the chart where the bulk storage tank would appear with relation to the plant as shown by Defendant's Exhibit 6?

A. The storage tank would be located on the margin of this diagram.

Q. Where would it be connected with the flow of pellets?

A. It would be connected to the diagram where the arrow is shown.

Q. It carries the words "To Storage"?

A. Yes, to storage.

Q. Will you please refer to the photograph which has been marked as Defendant's Exhibit No. 8 and tell us about that?

A. Photograph No. 8 shows the interior of the mixer drums before they are completely assembled. It shows the interior of the drum before the end plate has been attached.

Q. Is that a drum which is the same as the drum you have indicated as the upper agitating and lower agitating drum?

A. They are both identical.

Mr. Smith: At this time we will offer in evidence Exhibits 7, 7-a, 7-b, 7-c, 7-d and 8, the latter being the picture which has just been referred to by the witness in his testimony.

(Defendant's Exhibits Nos. 7, 7-a, 7-b, 7-c, 7-d and 8, filed.)

Q. Can you state approximately when the defendants Sayre pelleting plant was put into commercial operation? [fol. 117] A. Yes, it was put into commercial operation about January 3, 1939. It was January 3 they actually started.

Q. I understood you to say before the noon recess that the defendants had other plants for forming these carbon black pellets which are similar to this Sayre plant. Will you please enumerate the plants?

A. By name?

Q. Yes.

A. We have one operation at the McElroy plant.

Q. State in each instance when those plants were put in operation, if you know.

A. The McElroy plant was put in operation in the fall of 1938.

By the Court:

Q. Where is it located?

A. At Borger, Texas.

Q. Where is the Sayre?

A. At Sayre, Okla. Then we have another plant at Sanford, Texas.

By Mr. Smith:

Q. When was that plant put in operation, if you know?

A. Last year. I do not know exactly the month.

Q. Do you mean 1940?

A. Yes. We have another plant at Stinnet, Texas, also placed in operation in 1940.

Q. Under what trade name or trade names is the product sold which is made at the defendants' Sayre plant?

A. The trade name is Dixie-Densed.

Q. Before the noon recess you stated that you maintained a proportion of black to water in the upper drum. I believe you stated that the black was approximately 35 to 40 parts by weight and the water was in the neighborhood of 60 to 65 parts by weight.

A. Yes.

Q. Within what limits are these percentages maintained in your commercial production?

A. They are maintained within the limits of 65 to 58, approximately, per cent of water by weight.

Q. And as to the black?

A. It would be 35% of black and 42% of black, approximately.

[fol. 118] Q. Are you acquainted with all the operations of the defendant companies for the manufacture of the product we have been referring to as dustless carbon black?

A. Yes, sir.

Q. Do you know of any operation of either of the defendant companies anywhere where the so-called dustless carbon black is formed by the two-liquid process?

A. No.

Q. Do you know of any operation of either of the defendant companies where any liquid other than water is used?

A. No, we use exclusively water.

Q. Is there any reason why water is used in the upper drum on those operations where the pellet- are formed by the wet process?

A. To facilitate agitation and the densifying of the pellets.

Q. Have you studied the Wiegand-Venuto patent which is here in suit?

A. Yes, I have.

Q. Are you familiar with the process for forming pellets which is described in that patent?

A. Only from a laboratory standpoint, I have never seen that process used commercially.

Q. Do you know of any commercial production of dustless process pellets by the two-liquid process disclosed in the Wiegand-Venuto patent?

A. No, I do not.

Q. Assuming that you should be asked to manufacture pellets of this dustless carbon black which would have a

pellet size of, let us say, one-quarter inch in diameter, could you make such a pellet in the defendant's Sayre plant?

A. Yes.

Q. What would be necessary to make such a pellet?

A. We would have to increase the proportion of water to black and slightly rearrange the spacing of the pins in the drum. The pins would have to be spaced a little farther apart. Then we would get a fairly large percentage of this agglomerate.

[fol. 119] Q. Assuming that you are requested to make a pellet which is much smaller than the present commercial pellet, could such a pellet be made in the defendant's Sayre pelleting plant?

A. Yes.

Q. Would you do the same things to get the smaller pellet?

A. We could make a smaller pellet with exactly the same arrangement as we now have except to lower the percentage of water used and increase the proportion of black.

Q. Would you make any change in the screen at the end of the drier?

A. Not unless it was necessary to manufacture a specified and defined size.

Q. Do you control the size by the screen at the end of the drier?

A. Yes; if we have to have a certain percentage of fixed diameter pellets.

Q. In the operation at the Sayre plant, as shown on the chart Defendants' Exhibit No. 6, I believe you stated that the loose black was used as it came from the burner house?

A. As it came from the burner house.

Q. Is there anything done to that black prior to the time it enters that upper agitating drum?

A. No, it is merely conveyed to a storage tank located above and to the left of those mixer drums.

Q. Is there any ash or other impurities of that type in the carbon black at the time it enters that upper agitating drum?

A. Only such ash or grit as is ordinarily produced with a channel black from the normal production.

Q. Is there anything done in the pelleting plant as shown in Defendant's Exhibit No. 6 for the removal of that ash or grit?

A. No, nothing done.

Q. Is there any control of the purity of the final product or the purity of the loose black that enters that upper agitating drum?

A. No, sir.

Q. Do you know when the operation of the defendant companies began in the making of this dustless carbon black [fol. 120] by what we term the "dry process"?

A. Yes, in October, 1934.

Q. And I believe you referred to that as the Cabot process?

A. That is what we call the Cabot process.

Q. Do you know whether or not this process corresponds to the processes of the so-called Cabot group which is included in the plaintiff's booklet Exhibit No. 1? It refers to the Billings 210,254, Billings 2,039,766 and 2,120,540, also 2,120,541, which is a Billings patent?

A. Yes.

Q. Do you know upon what basis the defendant companies used this process under these patents?

A. We have an operating agreement with Cabot for using the Cabot patent.

Q. Do you know whether or not that includes a patent license agreement?

A. Yes, it does.

Q. Will you please refer to the process that you have described as the "extrusion process"? Do you know when the defendant companies began the operation of the extrusion process?

A. We began operations in 1934.

Q. Is that operation still continuing?

A. Yes, sir.

Q. Is the operation under the dry agitation process also still continuing?

A. Yes, sir.

Q. As chief engineer for the defendant companies, if you were asked to consider the process of the Wiegand-Venuto patent as a process for possible commercial production of pellets would you, or would you not, recommend the adoption of such process?

A. I would not recommend the adoption of that process.

Q. Why would you not recommend the adoption of the process?

A. I would consider that process impracticable.

Q. From a commercial standpoint?

A. From an operating standpoint.

Q. Would you care to elaborate on that?

A. First that process would of necessity have to be a batch [fol. 121] process. I think it could be made continuous by more elaborate equipment, but as the operation is described in the patent, I would get the impression that the process would be a batch process. Then use of a liquid such as gasoline I would consider very wasteful and it would be very difficult to recover the gasoline after the operation has been performed. For those reasons I would consider the process commercially impracticable.

Q. In connection with your work in designing the Sayre dustless plant of the defendants do you consider the requirements of the purchasers of your product in laying out those plants?

A. Yes.

Q. Will you state briefly what requirements the purchasers of your products specify, particularly first as to pellet size.

A. As far as I know there are no specifications covering pellet size. The primary requirement was that the pellets should withstand handling to the consumer's plant and in the consumer's plant, so that the pellets during transportation in handling and in the consumer's plant will not break up into dust to the extent that the black will not flow freely to the conveying and weighing equipment there.

Q. Do you know of any specifications which require that the pellets have a spongy and porous interior?

A. No.

Q. Do you know of any requirement or specification which requires that the pellets have some commercial uniformity of size?

A. No.

Q. Do you know of any specifications which requires a carbon black pellet which is approximately 1/16 inch in diameter?

A. No.

Q. Approximately what is the density or apparent bulk of the pellets which are made at the defendant's Sayre pelleting plant, if you know?

A. The apparent density at the plant is approximately 24 and 25 pounds per cubic foot.

[fol. 122] Q. Approximately what are the apparent den-

sities of the carbon black pellets which are made by the extrusion process and sold by the defendants?

A. The apparent density ranges between 24 and 26 pounds per cubic foot.

Q. What is the density of the product made by the dry process?

A. Approximately 27 pounds per cubic foot.

Q. In connection with your work as chief engineer for the defendants, in the carbon black field have you had occasion to visit any rubber plant recently?

A. Yes. In the latter part of October, 1940, I visited the General Tire & Rubber Company plant at Akron, Ohio.

Q. Was the General Tire & Rubber Company using the so-called dustless black in its plant?

A. No, they used the compressed type of black.

Q. Is that what you have referred to here as the heavy compressed black?

A. Yes.

Q. Did you see any operation where that black was compounded into rubber stock at the General Tire & Rubber Company plant?

A. Yes, sir.

Q. Will you describe that briefly for the Court, please?

A. In that plant they had three Banbury mixers, one large and two smaller ones. The large Banbury mixer was enclosed, I would say, within three walls inside of the factory and the two smaller ones were enclosed within another space, three walls, within the factory. While I was there I witnessed the incorporation of black into rubber using all three mixers.

Q. Have you ever had occasion to visit a rubber plant that used the so-called dustless carbon black in Banbury mills for the milling of rubber?

A. Yes, several.

Q. Approximately when was your last visit to one of these plants?

A. I cannot recall the month, I believe it was 1940, I visited the Firestone plant.

Q. Were they using a so-called dustless plan in the Banbury mill operation?

A. Yes.

[fol. 123] Q. Did you have occasion to observe the milling of the dustless carbon black into the Banbury at the Firestone plant?

A. Yes.

Q. To the rubber in the plant?

A. Yes.

Q. Would you care to state how that operation compared as far as dustiness is concerned with the operation you observed at the General Rubber plant where the compressed black was used?

A. Of course at the General plant the black was introduced into the Banbury mixers by hand, dumping the black from the bags into the Banbury mixers. At the Firestone plant the dustless black was handled through their conveyor and automatic weighing system into the Banbury. The black was automatically weighed in certain pre-determined volumes and then dumped into the Banbury mixers. At no time is the black dumped by hand at the Firestone plant, where they have the Banbury mixers.

Q. What is the condition regarding the general cleanliness of the conveyor system at the Firestone plant?

A. As far as the conveyor system is concerned that is, of course, very clean compared with handling black in bags. But around the Banbury, nevertheless you find carbon black dust flying about.

Q. Is it dust to such an extent that precautions are taken in the plant in connection with it?

A. I would say there are no special precautions taken as far as the dustless carbon black is concerned. They had a dust removing system installed there prior to using the dustless black. That dust removal system is still in operation.

Cross-examination.

Mr. McLean: I want to note on the record that the product which is Plaintiff's Exhibit No. 20 is the product of the Sayre plant which has been described at some length.

Q. Mr. Hanson, do you know how much of the products of this extrusion process has been made and sold year by [fol. 124] year by the United Carbon Company?

A. I do not have the figures.

Q. Would they be available?

A. Yes.

Q. Would you get them, please? You stated that one requirement that your customers have with respect to this pelleted product is that it stands shipment without breaking

down and forming so much dust as to interfere with handling, is that correct?

A. Yes.

Q. Do they also want a product of such nature that it will disperse in the rubber?

A. Yes, of course.

Q. Do they have any requirements as to purity?

A. Yes, as far as the carbon black itself is concerned, but not as far as the pellets are concerned.

Q. Do you mean to say they will take pellets with any amount of impurities in them?

A. No.

Q. They do have requirements as to impurities?

A. Yes.

Q. They say that only a certain proportion of impurities will be in the product they buy, is that right?

A. They specify a carbon black must pass a 325 mesh screen within certain percentages.

By the Court:

Q. That has nothing to do with purity, has it?

A. So far as the purity of the carbon black itself, I have no knowledge of any specifications.

By Mr. McLean:

Q. When you refer to an 325 mesh screen you mean that the manufacturers insist that there be no particle, for example of grit, of such size that they will not go through a 325 mesh screen?

A. That is what I meant.

Q. Whatever requirements as to purity are specified your products meet?

A. Yes.

Q. This drawing, which is Defendants' Exhibit No. 6, is intended to be a diagrammatical portrayal of your plant, is that correct?

A. That is correct.

Q. Is it drawn to scale?

A. No, I do not think so.

[fol. 125] Q. As I recall you stated that one of these cylinders is 12 feet long?

A. Approximately.

Q. And 4 feet in diameter?

A. Yes.

Q. And if this were drawn to scale it would actually either look longer or narrower, would it not?

A. Judging from here I should say it should be slightly longer.

Q. In the actual plant this second cylinder, instead of being to the right of the first, is actually positioned directly under it, is it not?

A. Directly below.

Q. The cylinder itself is provided with a number of inwardly projecting pins such as are shown in Defendants' Exhibit No. 8, is it not?

A. That is correct.

Q. Those pins are arranged in rows going around the cylinder, not in the cylinder?

A. In the cylinder itself; going straight through, actually straight.

Q. How far apart are they spaced?

A. The rows are spaced three inches apart.

Q. How many rows are there in the cylinder?

A. Twenty.

Q. Spaced three inches apart?

A. I mean to say there are 45 rows actually.

Q. There are 45 rows in the line?

A. Yes, 20 around the circumference.

Q. And they are spaced three inches apart along the line, is that right?

A. That is correct.

Q. So that if this, instead of a diagram, were intended to be an actual drawing there would be no more of these pins showing?

A. No.

Q. In between each of these pins attached to the cylinder there would be a pin extending from this shaft?

A. Not between every one. The pins on the shaft are fastened single file.

Q. How far apart are they?

A. They are three inches apart axially.

[fol. 126] Q. Then between each row of pins axially there is one pin on the shaft which rotates around between them?

A. That is right, in the upper drum only.

Q. The lower drum itself is exactly the same, is that correct?

A. Identically the same.

Q. This product which passes through the 14 mesh screen is the product that goes to your storage bin and is sold?

A. That is the finished product going into the storage room.

Q. Just to make the record clear, in the real apparatus the second cylinder would go from right to left instead of from left to right?

A. That would be correct.

Q. Are you familiar with the Grote Patent RE-21,390?

A. Yes.

Q. It appears as part of Plaintiff's Exhibit No. 1 and belongs to the United Carbon Company?

A. Yes.

Q. Do you use the process described in that patent?

A. No.

Q. Did you ever use it any?

A. In a semi-commercial small scale operation.

Q. You never used it on a commercial scale?

A. No.

Q. Do you know why this procedure is not used?

A. The mechanical equipment would have been too costly to maintain.

Q. It is commercially impracticable because of high cost?

A. Yes, sir • • • [194-244].

• • • Testimony of Isaac Drogin • • • [246].

• • • Direct examination • • • [246].

• • • Q. Your name is Isaac Drogin?

A. Yes, sir.

Q. Will you state your residence and occupation?

A. I reside in Charleston, W. Va. I am Director of Research for the United Carbon Company.

[fol. 127] Q. How long have you been Director of Research for the United Carbon Company?

A. Since December 1, 1939.

Q. Does that also include some work for the defendant United Carbon Company, Inc., the other defendant in this case?

A. Yes.

Q. Will you please describe the nature of your work as Director of Research for the defendant companies?

A. The nature of my work for the past year has been to direct the chemical research pertaining to the development of carbon black, to find new uses for carbon black and improvements in production and likewise the quality of carbon black.

Q. Prior to becoming Director of Research for the defendant companies will you please state what work you have been doing?

A. Prior to joining the United Carbon Company I was connected with the J. M. Huber Company from 1922 to the end of November, 1939.

Q. Prior to May, 1922, had you had any experience in the field of carbon black?

A. No.

Q. During the period that you were connected with the J. M. Huber Company did you have any experience either with the manufacture or use of carbon black?

A. Yes.

Q. Will you please state what that experience was?

A. From 1922 to 1930 I was in charge of production of carbon black for J. M. Huber, where they had plants at Louisiana, Texas and Wyoming. From 1930 to 1939 I was chief chemist of J. M. Huber, Inc. with headquarters in New York.

Q. Is there a difference between the two companies that you mentioned in your previous answer?

A. None whatever.

Q. What was your work as chief chemist during the period you were in New York?

A. My work dealt chiefly with control testing pertinent to carbon black and research pertaining to carbon black [fol. 128] and a good deal of contact between J. M. Huber, Inc. and the companies to whom they sold carbon black.

Q. Will you please state the kinds of business with which you had contact where carbon black was used?

A. Practically all rubber companies.

Q. Any ink companies?

A. Yes, the J. M. Huber Company itself.

Q. Any other companies which used carbon blacks?

A. Hardly worth mentioning.

Q. Will you please outline your educational qualifications for the work you have been doing since 1922 in the carbon black field, making them as brief as possible.

A. I received my college education at the College of the City of New York, in New York, where I graduated in 1913. After a year of post graduate work in the chemical department of the college I became a research fellow at the Mellon Institute in 1914, where I was until 1921. During that time I gained my Master's degree and Doctor of Philosophy degree through the University of Pittsburgh, with whom the Mellon Institute has a connection.

Q. Was your Master's degree received from the University of Pittsburgh also?

A. Yes.

Q. What was the general nature of the educational work which you pursued at those two institutions?

A. It dealt with the study of physical chemistry and some organic chemistry. May I add that I also had a fellowship at the Mellon Institute pertaining to the distillation of gasoline.

Q. In connection with your work as Director of Research for the defendant companies have you studied their so-called dustless product which is made at the Sayre, Oklahoma, plant and sold as Dixie Densed carbon black?

A. Yes.

Q. Will you please state what tests you have applied and studies you have given to this product, particularly first with regard to size?

[fol. 129] A. When examining a sample of our Sayre production for size we are in the habit of taking a definite weight. Roughly, for our work we always take a 200 gram sample and screen it first through a 14 mesh sieve.

Q. Approximately what size of pellets will pass a 14 mesh sieve?

A. Any pellet that is smaller than .0555 inches.

Q. Do you know about what size that would be in terms of fractional inch measurement; would it be 1/16 inch or smaller?

A. Smaller.

Q. Approximately how much smaller?

A. 1/10 inch would, roughly, 1/16 inch would be, roughly, .06 inch, whereas the openings of a 14 mesh sieve are .0555. After we establish that all the pellets that were smaller than .0555 went through the particular 14 mesh sieve we took the pellets that went through the 14 mesh and screened them in a 20 mesh sieve.

Q. Do you have any records showing the result of your screening test operations of the product of the defendants Sayre plant?

A. Yes.

Q. Will you please produce such figures?

Mr. Smith: At this time we would like to offer in evidence as "Defendants' Exhibit No. 9," a folder which contains the table of U. S. standard sizes which the witness referred to in making his last answer.

(Defendant's Exhibit No. 9, filed.)

By Mr. McLean:

Q. Are these the same things we find in any handbook?

A. Yes.

The Witness: I found that 47% of our material went through a 14 mesh sieve and was retained on a 20 mesh sieve.

By the Court:

Q. Exactly 47%?

A. 47.1%. There were no pellets retained on the 14 mesh.

By Mr. Smith:

Q. What does that indicate to you as to the size of those pellets?

A. In the first place, that they were smaller than .0555 inches.

[fol. 130] Q. You mean, larger than what?

A. Larger than .0555 inches.

Q. Passing a 14 mesh screen indicated they were smaller than .0555 inches?

A. Yes.

Q. Pellets that were retained on the 20 mesh were of what size?

A. The pellets that went through the 14—they were smaller than .0555.

Q. That constituted 47% by weight of the sample?

A. Yes, of the sample that we took for examination.

Q. Will you please continue with your screening test figures?

A. We continued our screening through the 20 mesh sieve and caught the material on a 30 mesh.

Q. What percentage of material did you catch on the 30 mesh?

A. 32.3.

Q. What would that indicate as to the size of the 32.3 percentage material?

A. That they were smaller than .0555 and larger than .0232.

Q. What did you find as to the remainder?

A. We screened the remainder on the 30 and caught that on the 40 mesh.

Q. What percent was caught on the 40 mesh?

A. 11.05.

Q. What did that indicate as to the size of the pellets?

A. They were smaller than .0232 but larger than .0165.

Q. Then what was done?

A. Then we screened it on a 60 mesh and obtained 5.5%.

Q. What would that indicate as to the pellet size?

A. That they were smaller than .0165 but larger than .0098.

Q. What was done with the remainder of the carbon black sample?

A. We screened that on a 100 mesh and found it contained 2.3%.

Q. What did that indicate as to size?

A. That they were smaller than .008 but larger than .0059.

[fol. 131] Q. Was there anything left to screen after that?

A. Yes, 1.3% went through the 100 mesh screen.

Q. What did that indicate?

A. That they were smaller than .0059 inches.

By the Court:

Q. When you say smaller than .0059 inches what does that fraction represent?

A. Diameter.

By Mr. Smith:

Q. These figures that you have referred to are figures which you have found in a chart which is a part of the folder, Exhibit No. 9, is that correct?

A. Yes.

Q. Do you know whether or not that is a standard chart or a standard table of mesh sizes for different types of commercial testing screens?

A. Yes.

Q. Is that a common test which is applied in the carbon black industry?

A. Yes.

Q. To determine the size of the dustless particles?

A. It is a common test applied by the manufacturers of carbon black. Not so much by the users of carbon black since the users are only interested in determining the amount of grit that would be screened through a 325 mesh.

Q. In connection with these figures as to sizes which you have just given, do they represent a single test run by you or have you run more than one test?

A. I have run more than one test.

Q. Would you say that the figures which you have given are representative of all the tests you have run on the product of the defendants' Sayre plant?

A. I would say they are typical.

Q. Would that apply to the pellets as they might be drawn from the bulk storage tank or would that apply to pellets which had been handled in conveyor systems, etc.?

A. That would apply to pellets as they are taken from the storage tank.

[fol. 132] Q. Have you in your work as Director of Research for the United Carbon Company ever had occasion to test the matter of purity of the pellets made at the defendants' Sayre plant?

A. Yes.

Q. Will you please describe the test or tests which you used in checking the purity of this product?

A. The tests were in the nature of determining the percentage of volatile matter, ash, grit, acetone extract, acidity and moisture.

Q. Are those all standard tests which are applied to carbon black products to determine their purity?

A. Yes.

Q. Did you run similar tests on the black before it went into the pelleting drums at the defendants' Sayre plant?

A. Yes.

Q. And you ran the same tests on the dustless product made at the same time and with the same black?

A. Yes.

Q. Will you please state what you found as regards the presence or absence of impurities in the pelleted prod-

uct with relation to the presence or absence of impurities in the loose black?

A. There was practically no difference as regards the impurities in the black before it was processed in the pellets and in the resulting pellets.

Q. Was there any difference as to ash or grit content?

A. No.

By the Court:

Q. What do you mean by "grit": is that a hard particle of carbon?

A. It is a hard particle of carbon. We consider grit as that part which will be retained on a 325 mesh.

Q. It is actually in chemical composition carbon?

A. Yes.

By Mr. Smith:

Q. Will you state what you mean by the ash content of the carbon?

A. The ash content may be such impurities as sand, metal or any foreign matter that is not part of the black itself or should not be part of it.

[fol. 133] Q. Is there any separation or attempt to separate that material from the loose carbon black that enters the upper agitating drum in the defendants' Sayre plant?

A. None unless passing it through a micro-pulverizer.

Q. What is that?

A. It is a system where the black is caused to pass through a sieve, and the black is driven through the sieve by means of a number of revolving plates operated at 6,000 to 7,000 r.p.m.

Q. Is that a common type of apparatus used in the carbon black industry?

A. Yes.

Q. Where is that located with reference to the defendants' Sayre pelleting plant?

A. It is located in the packing house.

Q. Is it part of the defendants' Sayre pelleting plant or, rather, operation?

A. No.

Q. Does it have any effect on the purity of the pellets as distinguished from the purity of the loose black?

A. No * * * [246-256].

• • • Q. Is the micro-pulverizer in the defendants' plant ever used on anything other than the loose carbon black?

A. It is used only on the loose black.

Q. Whatever effect that may have on the ash or grit particles in the carbon black is completed by the time it goes to the pelleting plant, is that correct?

A. That is right.

Q. Does the micro-pulverizer take anything out of the loose black which passes through it?

A. Not very much.

Q. What does it take out of the loose black?

A. It really does not take anything at all. I would like to explain that. The micro-pulverizers are cleaned periodically and hard particles of grit which had not been pushed through the screen, if they are found on the edges or on the side of the micro-pulverizer are cleaned out. They are cleaned periodically in order to make sure that no blades are out of order.

[fol. 134] Q. What happens to whatever ash, grit or other impurities of that type may be in it when it passes through the micro-pulverizer?

A. It goes to the main product.

Q. Is there any change as to the size of the materials?

A. No, except the density of the bulk is reduced to about two or two and a half pounds per cubic foot.

By the Court:

Q. From what?

A. It might come from three. By the time it reaches the pulverizer it may be three pounds. The fluffy action fluffs up the black.

Q. You referred to ash: what is that?

A. It might be silica or metal scraped off the channels or through the passage of the carbon black into the conveyers.

By Mr. Smith:

Q. What happens to the ash and grit in the loose pulverizer?

A. They are left in the black.

Q. Then the micro-pulverizer does nothing in the way of purifying the black?

A. No.

Q. What other tests have you made in connection with the defendants' Dixie Densed pellets made at the Sayre plant?

A. I said we determined the chemical tests of volatile matter, acidity and moisture to determine the weight.

Q. What did you find in connection with the weight of these pellets?

A. Well, for instance, the apparent density. We found it to be 25.4 pounds per cubic foot.

Q. Was that on the basis of several tests or was that on the basis of a single test?

A. Based on several tests.

Q. That is actually the average figure?

A. That is an average figure.

Q. What other tests did you make on the product?

A. I determined the effect of the picking up of any organic liquid.

Q. What was the purpose of that test?

A. That test was to compare, I would say, the internal structure or internal density of that black against others.

[fol. 135] Q. To determine if it be spongy or porous?

A. Yes.

Q. Will you please describe that test?

A. Briefly, it consists of taking a pellet of uniform size——

Q. (Interposing) How do you get such a pellet?

A. By screening it. We screen it through an 18 mesh and we changed it on a 20 mesh.

Q. That would give you pellets of approximately what size?

A. Sizes between .0394 and .0331.

Q. Continue further and describe the tests.

A. We have a special tube; that is graduated. We fill that tube to a certain height with xylol, an organic liquid. Then we take a definite volume of these pellets and gradually pour them into this column of xylol and at the end of fifteen minutes note the height of the column of liquid.

Q. What does that indicate as regards the sponginess or porosity of these pellets?

A. If the column of liquid did not rise sufficiently high then it indicated to me that the internal structure of the pellets was such that they would drink up the liquid. If, on the other hand, the column of liquid rose above a certain

height it would indicate to me that the pellets were not porous and could not drink up the liquid.

Q. What was the result of that test so far as these products of the defendants' Sayre pelleting plant was concerned?

A. I found that as a result of adding this definite volume of pellet to the liquid the increase in height of the original column of liquid was 16.8 per cent.

Q. Will you please state how you determined this 16.8 per cent?

A. I assumed the original height to be, say, 25 cc. and when I added——

Q. (Interposing) That is, the original height of the liquid?

A. Yes, plus a certain other fraction. That other fraction was consistent in all my tests, so I considered 25 cc. as the height of the column of liquid alone. After I added the pellet the height of the column was 29.2 cc. and the average height was 29.17.

[fol. 136] Q. That is the average of what,—of several tests?

A. The average of several tests.

By the Court:

Q. An average rise of 16.8 per cent?

A. Yes.

Q. Does that mean they were thirsty?

A. They were somewhat thirsty. If I took pellets which I assumed were not porous at all then the height of the liquid would have been 32.8. Over 25, there would be an increase of practically 8/25 or 1/3.

Q. Did you apply that same test to a product such as that shown in Plaintiff's Exhibit 11, entitled the "two-liquid process?"

A. I could not apply that test to a pellet of that type.

Q. Why were you unable to test a pellet of that type?

A. Because I could not buy any pellet of that type made by the two-liquid process * * * [257-262].

* * * Q. Did you make an effort to obtain such a product on the market?

A. Yes.

Q. Failing to get such a product on the market, what did you do?

A. I made some myself.

Q. And will you please describe how you made pellets by the two-liquid process?

A. I made these pellets according to the disclosures in the Wiegand and Venuto patent No. 1,889,429 * * * [266].

* * * Q. Dr. Drogin, prior to the recess you stated that you had made pellets by the process, the Wiegand and Venuto patent No. 1,889,429.

A. Yes.

Q. Will you please describe how you made such pellets, stating as you do so the proportions of carbon black, water and other liquids which were used?

A. I made, or rather, I prepared several batches according to the disclosures of the Wiegand and Venuto patent.

Q. Now, just how did you do that?

A. I mixed carbon black and water until I had the black thoroughly wetted.

[fol. 137] Q. And do you have anything here in court that would show what you mean by carbon black being thoroughly wetted, at this stage of the process?

A. Yes.

Q. Will you please produce it?

A. I have here a jar representing the consistency of the mixture which I prepared when I wetted the black with the water.

Q. How much black and how much water was used?

A. In the particular preparation I used, 305 parts of black and 4,480 parts of water by weight.

Q. Approximately what would that reduce down to in the terms of the patent, which specifies carbon black 5, water 40 to 80 and gasoline 7 to 9?

A. That would be 5 parts of black to 80 parts of water. And I think that you can see and hear (shaking jar) that it is an extremely thin mix.

Q. Now, I would like to interrupt at this time to ask you to produce, if you can, a specimen which indicates the consistency of the mix used in the defendant's Sayre operation, please, Dr. Drogin.

A. I have here a mix consisting of, roughly, two parts of water and one part of black.

Q. And how does that compare with the 1 to 16?

A. There is really no comparison as far as fluidity is concerned. This is a crumbly mass. This is a liquid, a very fluid mix.

Q. That is the 1 to 16?

A. That is the 1 to 16, yes.

Mr. Smith: At this time I would like to have the bottle containing the 1 to 16 mix marked Defendant's Exhibit No. 10 and the bottle which the witness has referred to, which contains the 2 to 1 mix of carbon black marked as Defendant's Exhibit No. 11.

Q. Now, with that digression, Dr. Drogin, will you continue to tell us what was done with the 1 to 16 mix of carbon black and water, or as stated in the Wiegand patent, the 5 to 80 parts of carbon black to water, in the making of these pellets by the two-liquid process?

[fol. 138] A. *To this thin slurry of black and water* I added 548 parts by weight of gasoline.

Q. And what does that correspond to in the language of the patent, which specifies gasoline 7 to 9?

A. That is 9 parts of gasoline, which make the mixture five, eighty and nine.

Q. And why did you select that particular proportion of five, eighty to nine?

A. Because as a result of my experiments I found that that particular proportion gave me pellets closer to the size in which the inventors specified they produced by their process.

Q. And that size was what?

A. That size was supposed to be 1/16th of an inch.

Q. You mentioned in your last answer many experiments you made. You mean by that, many experiments in connection with the Wiegand and Venuto process?

A. Yes.

Q. Now, will you please continue and tell us just how you made these pellets?

A. Yes. Let me show you the mixer first. This represents a Daisy Churn. It is model No. 80, and the one that I used was similar to this particular sample except that the paddles or the shaft of the paddles was connected to a drill press so that I could rotate it much faster than I could by hand, and at the same time do the agitation or perform the agitation in a uniform manner every time I tried to make a mix, and by using this type of churn I obtained pellets in five minutes agitation.

Q. At what speed?

A. 755 r.p.m.

Mr. Smith: The churn which the witness has just referred to is now offered in evidence as Defendant's Exhibit No. 12.

(Defendant's Exhibit No. 12, filed.)

The Witness: After these pellets were formed and the water was clear and free from carbon, I skimmed off the pellets with a ladle strainer, put them in a glass dish and [fol. 139] dried them overnight; in fact, for 20 hours, at a temperature not exceeding 125 degrees Centigrade, and as measured in the black itself.

Q. Now, you observed the test or demonstration which was run here yesterday in connection with this Wiegand and Venuto two-liquid process, and you observed the initial step of mixing the carbon black with water, is that right?

A. Yes.

Q. Now, was that done by you in this churn or was it done in a separate container?

A. The mixing of the black and water was done by me in this churn, or done under my supervision.

Q. Then where was the gasoline added?

A. The gasoline was added through the top opening of the churn.

Q. Now, so far as the separation and the flotation of the pellets was concerned, did you secure much the same sort of separation and flotation that was secured in the demonstration apparatus here yesterday?

A. To a degree, yes, but much better looking.

Q. Can you produce the pellets which you made that you have just testified about?

A. Yes.

Q. Will you please do so?

A. Yes, here they are.

Mr. Smith: I will ask to have the specimen the witness has just produced marked for identification Defendant's Exhibit No. 13.

Q. Now, Dr. Drogin, referring to this specimen which you have produced, and which we have marked for identification Defendant's Exhibit No. 13, will you please state what is inside that container? By that I mean, are those screened or unscreened pellets?

A. These are unscreened pellets.

Q. Are they in the same condition that they were in when they came from the drying operation?

A. I would say almost, unless they got broken down in the course of handling.

[fol. 140] Mr. Smith: The container which Dr. Drogin has produced, which contains the specimen heretofore marked for identification, is now offered in evidence.

(Defendant's Exhibit No. 13, filed.)

Q. Now, did you subject the product which you mentioned heretofore as having manufactured according to the Wiegand and Venuto Patent No. 1,889,429, to the same physical test that you subjected the defendant's Sayre product?

A. Yes.

Q. Will you please state what you found in this connection, starting first with the sizes of the pellets, if you made such determination?

A. I found that when 200 grams of these pellets were subjected to a screen test 24.2 per cent remained on the 14 mesh.

Q. That would indicate what as to the size of those pellets?

A. That would indicate that they were larger than .0555 inches.

Q. In connection with the screening test, did you retain any specimens of the different fractions of these pellets which you screened?

A. Yes.

Q. Will you produce these fractions.

A. Yes.

Mr. Smith: I ask that these bottles just produced by the witness be marked for identification as Defendant's Exhibits Nos. 14 to 19, inclusive.

Q. Dr. Drogin, I hand you a sample bottle which has just been marked for identification as Defendant's Exhibit No. 14, and ask you if you will state what that is and what it contains.

A. This contains pellets retained on 14 mesh screen. These pellets came from the batch made according to the Wiegand and Venuto process, using the ratio of 5 parts of black to 80 parts water and 9 parts gasoline.

Q. And is that the fraction which is taken from a composite sample, such, for example, as that shown in Defendant's Exhibit No. 13?

A. Yes.

[fol. 141] Q. I now hand you a bottle which has been marked for identification as Defendant's Exhibit No. 15, and I will ask you the same question.

A. These are pellets representing 53.6 per cent. retained on a 20-mesh screen.

Q. That then would be the pellets which would pass the 14-mesh screen and were retained on the 20-mesh, is that correct?

A. Yes.

Q. And approximately what is the size of those pellets?

A. Those pellets are smaller than .0555 and larger than .0331.

Q. Now I hand you a container which has been marked for identification Defendant's Exhibit No. 16, and ask you the same question.

A. These are pellets which passed through the 20-mesh screen and were retained on the 30. They represent 9.2 per cent. of the composite which I tested.

Q. And what would be the approximate size of such pellets?

A. These would be smaller than .0331 and larger than .0232.

Q. I now hand you a container marked for identification Defendant's Exhibit No. 17, and ask you the same question.

A. This contains pellets that were passed through a 30-mesh and were retained on a 40-mesh screen. They represent 3.02 per cent.

Q. And what size pellets are represented in that container, Defendant's Exhibit No. 17?

A. These represent pellets smaller than .0232 and larger than .0165.

Q. I now hand you a container marked for identification Defendant's Exhibit No. 18, and I will ask you the same question concerning that.

A. These contain pellets which went through a 40-mesh screen and were retained on a 60-mesh. They represent 2.0 per cent. of the composite.

Q. And what size pellets are represented in that sample, Dr. Drogin?

A. These represent pellets smaller than .0165 but larger than .0098.

[fol. 142] Q. I now hand you a container marked for identification Defendant's Exhibit No. 19, and ask you the same question.

A. These represent pellets that went through this 60-mesh and were retained on 100-mesh.

Q. And what percentage of the total was that?

A. 6.2 per cent.

Q. And what size particles would be represented in this container?

A. They would be particles smaller than .0098 but larger than .0059.

Mr. Smith: At this time we will offer in evidence as Defendant's Exhibits Nos. 14 to 19 inclusive the containers heretofore referred to.

(Defendant's Exhibits Nos. 14 to 19, filed.)

Q. Now, in connection with the test as to particle size which you ran on these pellets, how large a sample of the pellets is represented in the large container, for example Exhibit No. 13?

A. I don't know what the actual weight is. I filled this display jar with sufficient quantity so as to come up near the top.

Q. I notice different levels in the smaller containers, Defendant's Exhibits 14 through 19, and ask you if those different levels represent anything.

A. They represent approximately the pellets that were obtained from a 200—I believe from a 200-gram sample of the composite.

Q. So that the level of the black in the small jars, Exhibit 14 through 19, gives some sort of a graphic representation of the difference in fractions?

A. They certainly do.

Q. Now, what other tests did you run in connection with the Wiegand and Venuto two-liquid pellets represented by that composite sample, Defendant's Exhibit No. 13?

A. I determined their bulk or their apparent density.

Q. And what did you find was the bulk or apparent density of this product?

A. 17.6 pounds per cubic foot.

[fol. 143] Q. And how did you make this determination?

A. According to the procedure described in the booklet distributed by the Binney & Smith Company.

Q. What is the name of that booklet, if you know, Dr. Drogin?

A. I think it is called "Colloidal Carbons." I can get you the name of the book, or describe the test, if you wish.

Q. Will you please describe the test?

A. The test consisted simply in filling a 100 cc. graduate cylinder to the 100 cc. mark. First you weigh the cylinder and then re-weigh it after the black reached the mark of 100 cc., and we proceeded to gently tap that cylinder until the black would no longer settle and then calculated the bulk or the pounds of black per cubic foot.

Q. What method did you employ in determining the bulk or apparent density of the sample of defendant's Sayre product, Dr. Drogin?

A. The same procedure.

Q. So that the comparison of the apparent density of the two-liquid pellets could or could not be made with the apparent density which you have given for the Sayre pellets on the basis of similarity of tests?

A. Since the tests were similar I would judge that the comparison should also be similar, too.

Q. Have you any reason to believe that the comparison is not accurate?

A. No, no reason at all • • • [266-278].

• • • Q. What inaccuracies might come into the two tests which would upset the comparison which you have made?

A. First, inaccuracies of weight; second, inaccuracies of reading the final volume of black in the cylinder, but we were very careful not to make such an error, not to make any errors along those lines, so I would judge that the test is comparable for any black that we examined.

Q. Did you make more than one test on the pellets which you made by the Wiegand and Venuto patent?

A. We made several tests.

[fol. 144] Q. And what did you find on these tests, so far as the apparent density was concerned?

A. Along the order that I indicated.

Q. How much variation did you find in those tests?

A. Very small variation. I would call those within experimental error, depending on how the final volume was read.

Q. Would it be an error as much as 5 pounds per cubic foot?

A. Oh, no.

Q. Would it be an error as much as 1 pound per cubic foot?

A. Not even that much.

Q. Would there be an error of 6/10 of 1 per cent?

A. Within less than half of 1 per cent.

Q. Less than half of 1 per cent?

A. Yes.

Q. What about the tests you ran to determine the apparent density of the defendant's Sayre product?

A. It was of the same procedure with the method I just described.

Q. What would you say would be the possibility of error in that test expressed mathematically?

A. The same: Less than half of 1 per cent.

Q. Did you run any other tests on the product which you made following the Wiegand and Venuto patent, such as that shown in Exhibit No. 13?

A. Yes, I also determined the ability of the pellets to drink up this organic liquid xylol.

Q. And what test did you use for making this determination, Dr. Drogin?

A. The same procedure I described for the Sayre product.

Q. And what did you find as the percentage of porosity, I believe that is what you term it?

A. Yes. In this case I found the increase was only 10.4 per cent., which indicated to me that the pellets were porous and naturally could take up more liquid than the Sayre product did.

[fol. 145] Q. Have you ever attempted to weigh individual pellets of the defendant's Sayre Dixie Densed product and the Wiegand and Venuto products, such as Exhibit No. 13?

A. Yes.

Q. Please describe what was done in this connection.

A. We took two screens, one with an opening of .0287 and the other with an opening of .0280, and we screened first the Sayre black, Dixie Densed. We did that in order to obtain a pellet of close uniform size as possible. Then we

took at random 100 of these screened pellets and weighed them.

Q. And what did you find that 100 of those screened pellets weighed?

A. The 100 screened pellets, the Dixie Densed pellets, weighed .0121 grams, whereas the Wiegand and Venuto pellets weighed .0098 grams.

Q. Now, how did you select the 100 Wiegand and Venuto pellets?

A. Also by first screening them on a screen with .0287 inch opening and retaining those that were left on the screen of measurement .0280.

Q. And how did you select the 100 pellets?

A. Took them at random.

Q. Did you make any other comparative tests of this Wiegand and Venuto product and the defendants' Sayre product?

A. I examined in a procedure of ours, or what we call the crushing strength of the pellet.

Q. Will you please describe that test briefly?

A. The idea is to take one pellet at a time and place it on a plate and let another plate fall on it gradually by means of pressure and noting the reading on a gauge as to how much air pressure is required to crush that pellet.

Q. And is air pressure used in moving that upper member also?

A. No.

Q. How is that done?

A. Let me describe that to you at a little bit further length, after I find my records.

Mr. Smith: While the witness is looking for his records, I would like to take this opportunity to offer in evidence [fol. 146] as Defendant's Exhibit No. 20, the publication entitled "Flow Carbon," and which carries the copyright notice, 1937 Binney & Smith Company * * * [278-282].

* * *. (Defendant's Exhibit No. 20, filed.) * * * [282].

* * * Q. I show you a booklet entitled "Flow Carbon," which has been offered in evidence as Defendant's Exhibit No. 20, and ask you if you have had occasion to read that book.

A. Yes, I have read it.

Q. I will direct your attention to page 28, chapter 5, the section entitled, "Structure of Micronex Beads," and ask you to read the second and third paragraphs on that page, please, Dr. Drogin.

A. (Reading) "In order to determine the inner structure of a pellet or bead it is obviously necessary to fracture it. However, if this is done mechanically there is danger of crushing with subsequent loss of structure. A practical means to accomplish fracture without crushing has been found to consist in covering the pellets with water, allowing them to settle and after decanting, placing the moist pellets in an oven to dry. This wetting and drying gently breaks the pellet into three or four pieces. Under a microscope these fragments clearly show the structure of the bead.

"An Outer Shell Enclosing a Porous Mass.

"Studied under the microscope (X28) it was found that the structure was made up of a distinct shell covering an inner mass, this body being porous in comparison with the shell. In some cases the shell had split into only two parts, disclosing a porous mass. There was no indication of any more than one shell enclosing the porous inner body. It is perhaps interesting to note that in a quiet room when the shell is pierced with a sharp instrument it cracks open with a very faint but distinctly audible click."

Q. Now, following the instructions contained in those two paragraphs, have you made any examination of defendant's Sayre pellet and the Wiegand and Venuto pellets [fol. 147] which you made, Dr. Drogin?

A. Yes, but I did not subject them to moistening with water first.

Q. And why did you omit this step?

A. Because I found as a result of many tests that I could easily fracture the pellet into fragments which would clearly disclose the characteristics of the interior portion of the pellet.

Q. Now, as a result of your work with these pellets in your study of these pellets in the microscope, will you please state what you found as regards the interior structure of the defendant's Dixie Densed pellet? Also as to the Wiegand and Venuto pellets which you made.

A. I was able to corroborate the description of the pellet as indicated in the Wiegand and Venuto patent. I found that the pellets consisted of a shell inclosing a porous inner body.

Q. Which pellet are you referring to when you say you found that structure?

A. In the Venuto and Wiegand pellet.

Q. Does that structure exist, or did you find that structure in defendant's Sayre pellet?

A. No.

Q. Will you please describe what structure you found in the defendant's Sayre pellet?

A. I found a much more dense interior portion.

Q. Would it be possible for you to show the Court the results of cracking a representative number of these pellets under the microscope?

A. I believe I can.

Mr. Smith: Would your Honor care to see what it looks like through the microscope?

The Court: I am willing to take a look at it, yes.

Mr. Smith: I don't care about the mesh. Let us take the same mesh of both products. Now, will you please state for the purpose of the record what you are doing at the present time?

[fol. 148] A. I am now engaged in placing several pellets of our Sayre product known as Dixie Densed——

Q. What size, please?

A. Retained on 20 mesh.

Q. And what did they pass through?

A. They passed through a 14 mesh, I believe.

Q. Is that bottle an exhibit?

A. I don't think so.

Mr. Smith: Well, in order that the record will be clear, we will offer as Defendant's Exhibit No. 21 the bottle and contents from which Dr. Drogin is taking the sample of defendants' Dixie Densed pellet.

(Defendant's Exhibit No. 21, filed.)

Q. Now, you are placing those pellets on what kind of slide?

A. I am placing the pellets on a microscopic slide which contains a single depression, and I am examining them at a magnification of about 28.

Q. On what kind of microscope?

A. A binocular microscope.

Q. Now, those were the pellets from this bottle, Exhibit No. 21?

A. Yes.

Mr. Smith: Would your Honor like to see a comparable picture of the Wiegand and Venuto pellets of the same size?

The Court: Yes, I believe I would.

By Mr. Smith:

Q. Will you please prepare such a slide?

A. Yes, sir.

Mr. Edmonds: May I ask what is being done here? Are you now putting on the slide some pellets as made by Dr. Drogin in accordance with his notion of the Wiegand and Venuto process?

Mr. Smith: That is correct. They are the same fractions of pellets which he used in connection with the defendant's Sayre product.

[fol. 149] Mr. Edmonds: When you finish doing that will you also put on some two-liquid process pellets as made by Mr. Wiegand, or under his direction?

Mr. Smith: We would be glad to.

Mr. Edmonds: We are quite satisfied that these two-liquid process pellets as made by Dr. Drogin were not made by the process approximating that described.

Mr. Smith: Well, if you will produce a specimen which is your idea of the two-liquid pellet, we will be glad to put them on the third slide so that the Court can have the benefit of viewing these pellets at the same time.

Mr. Edmonds: I will now ask Dr. Drogin to put a specimen of the pellets which have been produced by the plaintiff as representative of the Wiegand and Venuto two-liquid pellet on a third slide to show these pellets to the Court, the pellets being taken from the sample which is in evidence as Plaintiff's Exhibit No. 11.

Mr. Smith: It is noted on the record that the sample, Plaintiff's Exhibit No. 11, represents an unscreened fraction of the pellets.

Q. Now, Dr. Drogin, I will ask you to examine each of the three slides and to state on the record what you observe as to the shape and general appearance of each product on each of the three slides.

A. I shall first examine the pellets from Exhibit No. 11.

Q. That is Plaintiff's Exhibit No. 11?

A. Yes, sir. I see before me practically all rounded pellets, very smooth surface. There are one or two which are larger in size than the rest. There are a few potato-shaped ones. The pellets have a shiny appearance, and also what I would consider bald like.

Q. Was that word "bald"?

A. (Spelling) B-a-l-d.

Q. Now, will you please examine the next slide and tell us what it is.

A. The next slide——

[fol. 150] Q. Particularly with regard to the characteristics which you have just commented on in the sample of the plaintiff's product.

A. I now have before me the pellets which I made according to the Wiegand and Venuto process. In all I see before me 10 pellets. Most of them are potato-shaped with a smooth surface, although in this particular group I find each pellet having a shell with a porous appearance.

Q. Now, will you please examine the slide of the defendant's Sayre pellet and describe what you see?

A. I see before me 16 pellets, some are spherical, none are—well, I will say one is potato-shaped. The surface is not as smooth as those that I had seen in the first slide I examined, which I believe was Exhibit No. 11.

Q. That is Plaintiff's Exhibit No. 11, the beads made by plaintiff's two-liquid process?

A. That is right. These pellets are not as finely—they are not as fine as those I saw in Plaintiff's Exhibit No. 11.

Q. What about the surface texture?

A. The surface texture is a little bit rough. It is not as smooth and velvety-like in appearance, rather a pitted condition.

Q. How does it compare with the, I believe you termed it, "bald" appearance of the first slide?

A. These definitely are not bald-like.

Q. Is there anything further in connection with the pellet that you observe in your examination.

A. These are quite different from the Wiegand and Venuto pellets that I made. They lack particularly the porosity of the shell.

Q. Now I will ask you to crack one of the defendant's Sayre pellets which is on your slide so as to reveal the internal structure of the pellet.

Mr. Edmonds: Are you going to do that over there?

A. Yes, I will do it right here.

Mr. Edmonds: Well, in this case I wish you would crack several samples so that we will see the variation, if there [fol. 151] is any variation, in internal structure in several of the pellets.

The Witness: I just broke one and it looked very nice.

By Mr. Smith:

Q. Will you please elaborate on what you mean by "very nice"?

A. I say it because it is quite a trick to break them without shattering them. This one, first of all, the fracture dense. It is dense through the interior, I mean. I see before me just one hole in the center, or two holes in the center of that product.

Q. What product is that?

A. That is the defendant's product.

Mr. Smith: Your Honor, Dr. Drogin has cracked one pellet of the defendant's product.

Q. Now, will you please take the slide which contains the Wiegand and Venuto two-liquid pellet from Plaintiff's Exhibit No. 11, and similarly crack a pellet or pellets so as to get a comparison with the pellet of defendant's product which you just cracked?

A. All right.

Q. Did you hear any audible click as those pellets were cracked?

A. That did not strike me, no.

Mr. Edmonds: May I point out on the record that room is quite noisy?

By Mr. Smith:

Q. Will you please describe what you see there in the cracked pellets which you have just cracked?

A. I have cracked a number of them and I find the fractures are clear and the internal structure shows the presence of considerable voids and unevenness, showing that the density of the interior is quite varying. I would consider that spongy and very porous.

Q. Now, will you please crack a few of the pellets on the third and remaining slide, which is the specimen of the Wiegand and Venuto pellets which you made? Is there any question about what the slide is you have just cracked the pellet on?

A. That is Plaintiff's Exhibit No. 11.

[fol. 152] Q. Now please perform the same operation on the pellets which you made.

A. All right. Here I find the fracture is clear and the interior of the pellets spongy and porous looking. The voids here are small but it definitely indicates porosity, or what we call a degree of sponginess * * * [283-292].

By Mr. Smith:

* * * Q. Now, Dr. Drogin, I would like to have you refer to page 29 of the booklet, "Flow Carbon," and particularly to the picture appearing on that sheet entitled, "Photomicrograph of split Micronex Bead showing internal structure," and I will ask you whether or not that is a picture which is representative of the internal structure of the Wiegand and Venuto two-liquid pellets, Plaintiff's Exhibit No. 11, which you have just cracked under the microscope.

A. The picture that I see before me represents fairly well the structure of a Wiegand and Venuto pellet which I just examined under the microscope.

Q. And how does that compare with the pellet which was made by defendant's Sayre process, in appearance?

A. This pellet which I see before me in no way resembles the interior of a Sayre Dixie Densed pellet. The picture before me shows what I construe as a large number of voids and difference in density, whereas the Sayre pellet which I just examined showed pretty closely uniform dense interior with only one hole.

Q. Now, Dr. Drogin, you have made a large number of different types of tests on the pellets which you made according to the Wiegand and Venuto patent and the Sayre dustless product. Would you care to comment on these two products as regards their relative porosity or sponginess?

A. From the tests that I have made I conclude that the pellets made by the Wiegand and Venuto process, the two-liquid process, those pellets are much more porous and spongy than the Dixie Densed pellets made in the Sayre plant.

Q. Have you ever made any tests which would indicate that the contrary were true?

A. In all my observations and examinations of the pellets [fol. 153] I found that the usual thing was to find a Dixie Densed pellet with a very dense or rather a dense interior in comparison with the spongy and porous appearance of the Wiegand and Venuto pellets made by the two-liquid process.

Q. In your microscopic study of the two pellets could you give us any idea of the approximate number of the pellets which you have studied under the microscope?

A. I could not state exactly the number but I have made a very large series of tests, and in each case I would have from 8 to 20 pellets on the slide, and I would generally break open a large number of the pellets on the slide which I was examining at the time, and my observations invariably were to the effect that he Wiegand and Venuto pellets made by the two-liquid process were spongy or porous, in line with what the inventors claimed in their patent.

Mr. Edmonds: Will you make that more clear? Now, by these Wiegand and Venuto pellets you mean pellets such as these you have made and that are in these exhibits before us?

The Court: I think that is perfectly clear, but right there let me ask a question:

Q. You have referred right along to Plaintiff's Exhibit No. 11 as a two-liquid process. Is that not, as a matter of fact, a one-liquid process bead made by the Glaxner process?

Mr. McLean: Your Honor, these beads in Exhibit No. 11 were made in the past few weeks in this machine which was operated here yesterday, only the machine didn't break down then.

The Court: And they are actually two-liquid beads?

Mr. McLean: They are actually beads made exactly in accordance with the way they were made in court yesterday.

Mr. Smith: I don't know whether that is clear or not, your Honor.

[fol. 154] The Court: Yes, I just wanted to be sure about that.

By Mr. Smith:

Q. Will you give us a general estimate as to how many of these pellets you have studied in this way? Would you say it was 10 or 100?

A. I would say near to thousands.

Q. And did you find anything in this examination of these pellets which would lead you to believe that the results which you just attained under the microscope over there were not typical results?

A. The results that I just pointed out are typical of the Wiegand and Venuto pellets and also of the observation that I made for the Dixie Densed pellets.

Q. Approximately how many of the defendant's Sayre pellets have you examined in a similar way, would you say?

A. I would say even more than those that I examined of the Wiegand and Venuto, because part of my work in the plant has been to study the characteristics of our product.

Q. Based upon this examination of a large number of pellets, would you say that the results you just achieved under the microscope over here were typical or not typical?

A. They are typical * * * [292-296].

* * * Mr. Smith: At this time we will offer in evidence Defendant's Exhibit No. 22, a folder of the patents which have been set up in our answer, and as Defendant's Exhibit No. 23, a booklet of patents which have been selected as showing the state of the art and are the patents which are enumerated in the notice which was served on plaintiff's counsel. I will hand these to your Honor, because we have other copies to use in the examination of this witness.

(Defendant's Exhibits Nos. 22 and 23, filed.)

Mr. Smith: Now, I think at this time—so that we will have the offering of the exhibits clear—that we will offer in evidence, as Defendant's Exhibit No. 24 a certified copy of the file wrapper and contents of the Wiegand and Venuto patent No. 1,889,429 * * * [297].

[fol. 155] * * * (Defendant's Exhibit No. 24, filed.) * * * [298].

* * * Q. Dr. Drogin, on yesterday you stated that you had split many of these defendant's Sayre pellets and had examined them under the microscope, and I believe you stated that the specimen you examined yesterday was

typical as to its internal structure to the many pellets of defendants' Sayre product which you had previously examined. Is that correct?

A. Yes.

Q. I hand you a photograph, which I have marked for identification as Defendant's Exhibit No. 25, and I will ask you if you will examine that photograph and state what it is, or what it shows.

A. This photograph indicates to me a pellet in which the interior is rather dense, and quite uniform in density throughout.

Q. What would you say as to whether that photograph is a typical representation of the interior structure of pellets which you have examined under the microscope?

A. This photograph is typical of the many pellets of the Sayre black which I have examined under the microscope.

Q. Is it your opinion that this photograph is as generally representative of the interior of the defendant's Sayre pellets as is the photograph on page 29 of the booklet "Flow Carbon", Defendant's Exhibit No. 20, is typical of the many samples of the product which you have made and examined as being made under the Wiegand and Venuto patent?

A. Yes.

Mr. Smith: The photograph Dr. Drogin has just referred to is offered in evidence as Defendant's Exhibit No. 25.

(Defendant's Exhibit No. 25, filed.)

Q. Now, Dr. Drogin, will you look at the photograph, Defendant's Exhibit No. 25, and state what that photograph in your opinion shows regarding the character of the interior of the defendants' Sayre pellets?

A. The interior of this pellet indicates to me that it is [fol. 156] rather uniform in density throughout and decidedly free from much porosity. I can see, however, one or two points which would indicate a void; but on the whole the whole appearance of the interior of this pellet is that it is uniform in density.

Q. How does the general character of the fracture of these defendant Sayre pellets compare with the general character of the fracture of the product which you made following the teachings of the Wiegand and Venuto patent?

A. There is quite a difference in the fracture.

Q. In what way?

A. The one pertaining to the Sayre pellet is a clear fracture, showing uniformity, whereas in the Wiegand and Venuto type pellet the fracture is not as clean and it shows irregularities.

Mr. McLean: Just to clear up the record as to what——

The Court: I think your point is well taken. I think the record should show that the witness is now testifying from the photograph of the fractured pellet appearing on page 29 of Defendant's Exhibit No. 20. You may proceed.

By Mr. Smith:

Q. I believe you testified yesterday, Dr. Drogin, that the photograph on page 29 of Defendant's Exhibit No. 20 was, in your opinion, typical of the large number of pellets, or typical of the appearance of the large number of pellets which you had made under the teachings of the Wiegand and Venuto patent. Am I correct in that?

A. Yes.

Q. Is the pellet which you have referred to, and which is shown on Defendant's Exhibit No. 25, the pellet which we have referred to in this litigation as the defendant's Dixie-Densed Sayre pellet?

A. Yes.

Q. And did you have that photograph made for the purposes of the record?

A. Yes.

Q. I hand you a photograph, which I have had marked for identification as Defendant's Exhibit No. 26, and ask [fol. 157] you if you will look at it and state what it is and what is shown thereon.

A. This photograph is a picture or a representation of the instrument we used for determining the crushing strength of pellets.

Q. And is this the instrument to which you made reference in your testimony yesterday?

A. Yes, sir.

Q. Now, will you please, with reference to photograph marked for identification Defendant's Exhibit No. 26, describe briefly the tests which were run to determine the relative or comparative crushing strengths of defendants'

Sayre Dixie-Densed product and the pellets which you made following the teachings of the Wiegand and Venuto patent?

A. Our tests simply consisted in placing the pellet on the top of the piston.

Q. Will you please place some reference numeral on the photograph to indicate what you mean by the piston; that is, on the photograph marked for identification Defendant's Exhibit No. 26?

The Court: Call that point "A".

A. All right; the pellet is placed at the point A, which represents the top of the piston.

By Mr. Smith:

Q. Is that a movable piston or a stationary piston?

A. It is a movable piston.

Q. All right; proceed.

A. By means of the rubber bulbs we gently——

Q. Will you mark the rubber bulb, please?

A. Yes, I will mark the rubber bulb point "B". We gently force air under the piston to cause it to move upward, and this is continued until the pellet touches the stationary anvil.

Q. Will you mark that with the reference letter "C", please?

A. I will mark that "C". Pressure is continued gently until we see the pellet crushed.

Q. Now, how is that pressure measured?

A. That pressure is measured on a sensitive pressure [fol. 158] instrument, which is similar to the ones that physicians use in determining the blood pressure of a person.

Q. And is that shown on this photograph?

A. Yes.

Q. Will you mark that with the letter "D"?

A. Yes. This picture does not show a pellet on there. Do you mind if I draw it on?

The Court: No; I think it is perfectly clear without that.

By Mr. Smith:

Q. Now, will you state how this instrument determines the hardness of the pellets under test, and what reading

or other record is taken, which is an indication of such hardness?

A. Naturally a pellet which is hard requires more pressure for it to be broken than a pellet which is not as hard, and the numerical readings are taken from the sensitive pressure gauge.

Q. Now, what is done in connection with the selection of pellets that are tested? Are they of a uniform size, or of different sizes?

A. They were of uniform size.

Q. And what size pellets did you select for the test which you ran to determine comparative hardness?

A. We selected pellets that went through a .0287 opening and were retained on a mesh of an opening .0280.

Q. Now, how many pellets would you test in order to arrive at a figure which, in your opinion, represents accurately the relative crushing strength of the pellets?

A. A large number.

Q. Can you say approximately how many?

A. From twenty-five to fifty.

Q. And then what was done after you had tested twenty-five or fifty pellets and had taken the readings as to their crushing strengths?

A. I averaged all the readings for that particular type pellets.

Q. Do you have available the average readings which you compiled as a result of these tests?

A. Yes.

[fol. 159] Q. Will you please give us that figure for the Sayre Dixie-Densed pellets? * * * [298-305].

* * * A. The Sayre pellet black, known as Dixie-Densed, gave us an average of 16.8.

Q. Will you give us the similar figure for the product which you made following the teachings of the Wiegand and Venuto patent?

A. For the Wiegand and Venuto pellet, made in the proportion of five of black, eighty of water and nine of gasoline, I obtained an average of 6.2.

By the Court:

Q. What do you mean by that; 6.2 what?

A. These, your Honor, are in terms of millimeters of mercury, the readings on the gauge.

By Mr. Smith:

Q. Now, what do those figures represent to you, Doctor, as to the relative hardness of these two pellets?

A. The figures indicate to me that the Dixie-Densed pellet is much harder than the Wiegand and Venuto pellet.

Mr. Smith: The photograph which the witness has just referred to in the foregoing testimony is now offered in evidence as Defendant's Exhibit No. 26.

(Defendant's Exhibit No. 26, filed.)

Q. In your opinion, Dr. Drogin, does the process by which the defendants' Dixie-Densed Sayre pellets are manufactured and the process which you used in making pellets according to the teachings of the Wiegand and Venuto patent, No. 1,889,429, affect the internal structure of the pellets?

A. Why, yes.

Q. Will you please elaborate on your answer?

A. The pellets made by the Wiegand and Venuto two-liquid process show an internal structure which is highly porous and ununiform in density and have a distinct shell which is more dense than the interior; whereas the Dixie-Densed pellets show an interior which is more uniform, much more uniform in density and very little sponginess or porosity.

[fol. 160] Q. What feature or features of the process of the Wiegand and Venuto patent would, in your opinion, account for this difference in porosity which you have just mentioned?

A. In the first place, there is a difference in the processes.

Q. Will you state what that difference is, in your opinion, that accounts for this difference in structure, or what those differences are, if there are more than one?

A. In the Wiegand and Venuto process you have a situation or condition where the water is expelled by the gasoline; whereas in the Sayre process we have merely an agglomeration and concretion of particles by means of the water.

Q. What is the function of the water which is used in the pellet operation in the Sayre plant in making Dixie-Densed pellets?

A. It has several functions.

Q. Will you please enumerate them?

A. In the first place, it is to remove partially the occluded gases and air entrained in the black; another function is to help to concrete and agglomerate the flocculent black.

Q. Are there any other features?

A. Another feature is to sort of bind or cement the particles together, which is similar to the concreting action I referred to a minute ago.

Q. Now, in your study of the Wiegand and Venuto patent, No. 1,889,429, what do you consider to be the function of the water in that process?

A. It is purely to wet thoroughly the particles of black.

Q. Do you find in the experiments in connection with the use of water according to the teachings of the Wiegand and Venuto patent, any concreting of the particles, due to the use of the water?

A. No.

Q. When do you first note the formation of pellets in the Wiegand and Vennto process as you carried it out?

A. Only after the gasoline has been added.

[fol. 161] Q. I hand you here a copy of the Wiegand and Venuto patent, and direct your attention to page 3, lines 97 to 100, which read as follows:

"The whole process of pellet formation depends upon the selective wetting power which the various solvents exert on the carbon black,"

and I ask you to state whether or not, in your opinion, the process of pellet formation at defendants' Sayre plant for the manufacture of the Dixie-Densed pellets depends in any way whatsoever upon the selective wetting power of a solvent on carbon black.

A. The process at Sayre does not depend upon the selective wetting power of a solvent on the black.

Q. What does the process at the Sayre plant depend upon for the formation of the pellet?

A. Merely upon the concretion and agglomeration of the black.

Q. By what means?

A. By means of water • • • [306-310].

• • • Q. When do you first observe pellet formation in the defendants' Sayre plant; upon the addition of water, or after there has been some agitation?

A. After there has been some agitation.

Q. Now, in your opinion, Dr. Drogin, would it be possible to manufacture in the defendants' Sayre plant, using the water and the mechanical agitation solely, a pellet having the same internal characteristics as the pellets which you made following the teachings of the Wiegand and Venuto patent? • • • [311].

• • • A. No, we could not make a Wiegand and Venuto type pellet at the Sayre plant according to the procedure we use there now.

By the Court:

Q. Will you elaborate on that a little bit? Why?

A. Because in the Wiegand and Venuto process it is essential that you have a second liquid which shows preferential wetting, in order to produce the internal characteristics of the pellets as is evidenced in the Wiegand and [fol. 162] Venuto pellet, whereas in the Sayre process we need only water and agitation. All we need is water and agitation • • • [311-312].

• • • Testimony of Edmund Billings

Direct Examination • • • [313].

• • • Q. What is your name, age, residence and occupation?

A. Edmund Billings, 42 years old, residence in Weston, Mass. I am Vice President of the Godfrey L. Cabot Corporation.

Q. What is the business of the Godfrey L. Cabot Corporation?

A. The production of carbon black and natural gas principally.

Q. How long have you been familiar with the business of the Godfrey L. Cabot Company?

A. Since 1923.

Q. Does the Godfrey L. Cabot Company make carbon black in pellet form?

A. Yes, sir.

Q. When and where was the first plant of the Godfrey L. Cabot Corporation for the manufacture of black in pellet form established?

A. In the fall of 1932 in one of the plants of the Cabot Company near Pampa, Texas.

Q. What was the capacity of that plant and how much carbon black did you make that year?

A. That first plant was relatively small; it was 20,000 pounds a day.

Q. What would that be in annual capacity?

A. About 7,000,000 pounds a year.

Q. When and where, if you know, was the first enclosed system for handling carbon black installed?

A. In the consumer's plant?

Q. In the consumer's plant.

A. In one of the plants of the Firestone Tire & Rubber Company in Akron, in the summer and fall of 1933.

[fol. 163] Q. Describe, just in a word, how the carbon black is handled in that plant.

A. It is delivered to the plant in tank cars which look very much like a hopper bottom coal car except it is water tight in the roof. That car has a bottom opening and through a conveyor system raises the black to the roof of the plant some two or three hundred feet to the roof and then it flows through a system of gravity down into the mill below.

Q. Who was the first to ship carbon black in tank cars?

A. My company, the Cabot Company.

Q. Will you give us the sales figures of the Cabot Company of pellet black for the years 1933 and on? • • • [313-315].

• • • A. In 1933, 8,000,000 pounds; in 1934, 23,000,000 pounds; in 1935, 39,000,000 pounds; in 1936, 62,000,000 pounds; in 1937, 58,000,000 pounds; in 1938, 45,000,000 pounds; in 1939, 73,000,000 pounds.

Q. Who designed and installed the enclosed system of handling carbon black at the Firestone plant?

A. My company.

Cross examination • • • [315]:

• • • Q. At the time you started the sale of carbon black did you charge for it at the same rate you charged for the unpelleted product?

A. The same price, yes, sir.

Q. And that continued along through the early years?

A. Yes, sir, until quite recently.

Q. When you installed the handling apparatus at the Firestone plant that you spoke of in 1933 your company paid for that, did it not?

A. Yes.

Q. And that was the instaliation in the plant of the Firestone, and in handling the product by Firestone that installation was paid for by you?

A. By us, yes.

Q. You were never reimbursed by that company except as you may have made profits out of the sales to Firestone?

A. That is right.

[fol. 164] Q. Similarly you went on after that and installed apparatus in the plants of other users at your expense?

A. That is a broad statement. We installed one other at our expense.

Q. The next one?

A. The next one.

Q. You are the patentee or one of the co-patentees of the Billings-Offutt patents that we have discussed?

A. Yes.

Q. They relate to methods and apparatus for making carbon black in pelleted form, do they?

A. Yes.

Q. Those patents have all been applied for and granted in or since 1932?

A. That is right.

Q. One of the important ones was applied for in 1932 and issued in 1938; do you recall that?

A. I do not know but it is a matter of record. If you say so, I will admit it.

Q. You recall in the specification of that patent you stated that these pellets had striking commercial advantages, do you recall that?

A. Yes.

Q. That was true then and is true now?

A. That is correct.

Q. As a matter of fact the introduction of pelleted carbon black into the business of supplying the rubber manufacturing industry effected what one might call an industrial revolution; that is true, is it not?

A. Yes.

Q. And you have so characterized it from time to time?

A. Yes.

Q. And your counsel has so characterized it in legal proceedings that you have been involved in; that is true, is it not?

A. Yes.

Q. One or more of your patents was involved in a suit down in Texas in 1939 or 1940, was it not?

A. Yes.

Q. And you testified?

A. Yes.

Q. And you and your counsel told the Court down there that from 1915 on there was a dust problem presented to [fol. 165] the rubber industry using carbon black in connection with that use, generally referred to as the "problem of dustiness"?

A. That is right.

Q. And you and your counsel told the Court that the best men in the industry during those years had applied their capacities to provide a solution for that problem but had failed to do it, did you not?

A. Generally speaking, that is correct.

Q. Their efforts all failed, you told the Court?

A. That is right.

Q. And because their efforts failed the suppliers of carbon black supplied their black to the rubber companies in the form of compressed black?

A. Yes.

Q. And while that may have in some measure ameliorated the dust condition, it did not dispose of that by any means?

A. That is right.

Q. And the opposite party to that suit was the Huber Company, was it not?

A. Yes.

Q. And the representatives of the Huber Company joined you in making all those representations to the Court, did they not? I do not mean they acquiesced in that, but they stated the same thing in substance to the Court, did they not?

A. I do not recall that there was any difference of opinion on that.

Q. On those representations the decision of the Court was rendered holding your patents and the Huber Company's

patent that were involved to be valid, but that neither process or apparatus infringed on the patent of the other: that was the outcome, was it not?

A. I do not think I am capable of characterizing the Court's opinion in so few words as you do • • • [315-319].

Redirect examination • • • [319-320].

• • • Q. In the trial of the Cabot-Huber case you testified that you included the Wiegand-Venuto patent among the failures to solve the dustless problem?

A. In effect, yes.

[fol. 166] Recross-examination • • • [320].

• • • Q. And you based that statement on your statement that the process described there was one which led to too high a cost for the production of the pellets?

A. No, that was only part of the reason. The product of that process, as I saw it commercially, did not answer the demand. From another angle, the cost of making the material was obviously high, and the two together produced a commercial failure.

Q. And you stated you had seen one sample of the product of that process?

A. No, I am sure I did not say that for I have seen more than one.

Q. At the time back in those early years you had seen one sample which did not appeal to your notion of what a pelleted product should be?

A. I saw several samples.

Q. In 1932?

A. Back in different years; it was prior to 1932.

Q. Did not you use the expression down there that this Wiegand-Venuto product that you saw looked to you like black scrambled eggs?

A. That is right, exactly.

Q. That you thought was a fitting characterization of the product which you saw?

A. Yes.

Q. Would you look at Plaintiff's Exhibit No. 11, please? Would you say that looks like black scrambled eggs?

A. No, that is a good sample.

Q. It meets all of the commercial requirements so far as you can tell?

A. So far as you can tell with a glance with your eye, yes.

Q. I want to ask you to look at another product which has not been offered here and which is in a can.

A. This is one of them.

Q. That is a good product?

A. That is a good product.

Q. There is no resemblance in it to scrambled eggs?

A. None whatever.

[fol. 167] Mr. Edmonds: I want to have this can shown to the witness marked as "Plaintiff's Exhibit No. 22."

(Plaintiff's Exhibit No. 22, filed.)

2nd Redirect examination • • • [320-322].

• • • Q. What did you mean by saying that the Wiegand-Venuto patent which you saw looked like black scrambled eggs? Will you describe further what you meant?

A. The samples I saw and referred to in court were a commercial shipment made in paper bags. I think I testified at that time, and if I did not I will now so testify, that I saw several samples of the Wiegand-Venuto product. Those samples that I saw that were in tin cans were a good product as far as I could tell. The sample I saw delivered in paper bags, which was then the customary way of shipping carbon black, had broken down so inside of the bag was a mixture of pellets and broken pellets and "scramble" was a good description. It was a mixture of carbon and dust.

2nd Recross-examination • • • [322-323].

• • • Q. Did you know the full history of those bags you spoke of from the time they left the plant until you saw them?

A. No • • • [323].

• • • Testimony of Isaac Drogin.

Direct examination (continued).

By Mr. Smith:

Q. Dr. Drogin, I hand you a sample bottle, which has been marked for identification, as Defendant's Exhibit No.

2, and which has previously been referred to in the testimony in this case, and ask you if you can identify it and will state what it is. The sample is marked with the tag of the United Carbon Co., Inc., sample No. 22.

A. This sample represents pelleted black made by granulation in the dry state.

[fol. 168] Q. And that would be a specimen of the product which has been referred to in this litigation as the Cabot product?

A. Yes.

Q. Or the product made by the Cabot process, rather?

A. Yes.

Q. Now, I hand you another container which has previously been referred to in this record as Defendant's Exhibit No. 3, and which carries a tag of the United Carbon Co., Inc., sample No. 27, and ask you to look at this and tell us what that is.

A. This bottle, marked Exhibit No. 3, sample No. 27, is a sample of pellet black, made by granulation in the wet state, and according to my records, it represents pelleted black made at our Sayre plant, and is a material from the discharge end of the drier before it passes through the vibratory screen.

Q. I now hand you a container which has been referred to previously in the record as Defendant's Exhibit No. 4, and which contains the tag of the United Carbon Co., Inc., sample No. 15, and ask you to examine this container and tell us what this contains.

A. Sample No. 15 represents pellet black made by granulation in wet state according to the teachings of Patent No. 1,286,024 by W. C. Knowlton and H. H. Hoffman.

Q. I hand you a container, which has been referred to previously in the record as Defendant's Exhibit No. 5, and which contains the United Carbon Co., Inc., tag of the designation, sample No. 28, and ask you if you will tell us what is in this container.

A. Sample No. 28 represents black granulated and extruded in the wet state. This particular black, according to my record, was made at United Carbon Company's Sanford B plant.

Q. And is that the black that has been referred to in this record as the extrusion black of the United Carbon Company?

A. Yes.

Mr. Smith: Your Honor, at this time we will offer in evidence as Defendant's Exhibits Nos. 2, 3, 4 and 5, the containers and contents which the witness has just identified.

[fol. 169] (Defendant's Exhibits Nos. 2, 3, 4 and 5, filed.)

Q. Dr. Drogin, you have previously stated that prior to your employment by the United Carbon Company you had been employed by the J. M. Huber Corporation; is that correct?

A. Yes.

Q. Are you familiar, as a result of this employment, with the process used by the J. M. Huber Company and the product which it produces and sells as dustless carbon black?

A. Yes.

Q. I call your attention to the Price patent, No. 2,127,137, which is one of the patents in the book of patents, Plaintiff's Exhibit No. 1, and I will ask you to look at that patent and state whether or not that is the apparatus which is used by the Huber Company in its commercial manufacture of its dustless carbon black product * * * [324-327].

* * * A. In viewing the sketch in this patent it strikes me as being quite similar to the one that is used commercially by the J. M. Huber Company.

By Mr. Smith:

Q. Are there any substantial differences that you note, that you would care to direct the attention of the Court to?

A. I notice that in the drawing there are shown wire screen material in certain of their compartments. I feel that in the commercial drum that was used by J. M. Huber there is no screen wire lining, but essentially the Huber apparatus is along the line indicated in this drawing. In other words, there is a horizontal drum divided in four compartments. In the first two we have a roller, in the front of this horizontal drum you have an opening for the inlet of carbon black and a similar opening in the other end of the drum for the outlet of the carbon black, and like openings in the partitions dividing the compartments.

Q. Now, will you please refer to page 2, column 2, lines 49 and 50, of this patent, where there is a description of the product?

The Court: That is the Price patent?

[fol. 170] Mr. Smith: Yes, your Honor, the Price patent, No. 2,127,137.

Q. Please read those lines, Dr. Drogin, and state whether or not in your opinion that is an accurate description of the Huber product.

A. (Reading) "They are subsequently separated into coherent polyhedral agglomerates of a size that can be handled without dusting. The process is continuous and, therefore, well adapted to quantity production, and separate preliminary or accessory treatments and their accompanying cost of equipment and labor are unnecessary. The product is of a substantially uniform standard of quality, and well adapted to 'flowing' without dusting, it is compact and easily handled without substantial loss by dispersion in air, and is readily dispersible or miscible with the types of ingredients with which it is ordinarily used."

Q. Would you say that that portion which you have just read was or was not descriptive of the Huber product?

A. This is descriptive of the Huber product.

Q. Do you know the apparent density of the defendant's dustless product which is referred to as the extruded black, such as that represented by Defendant's Exhibit No. 5?

A. Yes.

Q. Will you please state what that density is?

A. The density, according to recent tests, ranges between 28 and 29½ pounds per cubic foot.

Q. Is this product a commercial product, sold commercially by the defendant company, that is?

A. Certainly it is.

Q. Who—just in a general way—uses this product? By that I mean what industries is this product used in?

A. It is used by the rubber industry and those who buy number among the largest consumers of carbon black for use in rubber.

Q. Have you studied the Geer patent, No. 1,245,700, a copy of which I now hand you?

A. Yes.

[fol. 171] Q. Will you please describe what you did in your studies of this patent?

A. I prepared pellet black according to the teachings of the Geer patent.

Q. Will you please describe how this was done, stating the proportions of carbon black and the liquid which was used?

A. I used a mixture of carbon black and kerosene in the ratio of two parts of black and one of kerosene.

Q. Do you find any mention of that particular ratio of black to kerosene in the Geer patent?

A. Yes.

Q. Will you please refer to the portion of the Geer patent where this ratio is given?

A. On the first page, the left-hand column, line 30 I read, "For example, I mixed two parts by weight of gas black with one part of kerosene oil in a suitable closed vessel".

Q. What did you use as a suitable closed vessel?

A. I used a Daisy churn as the vessel for preparing the pellet black according to the mixture which I just stated.

Q. And is the Daisy churn which you used similar to this churn, Defendant's Exhibit No. 12?

A. Yes.

Q. Do you recall how long you agitated the black and kerosene in the Daisy churn?

A. Yes.

Q. Will you please state how long it was agitated, Dr. Drogin?

A. Thirty minutes.

Q. And do you recall what the speed of the agitation was?

A. 755 r.p.m.

Q. I hand you a container which has on it the label of United Carbon Co., Inc., sample No. 23, and ask you if you can tell us what that is.

A. Sample No. 23 is black granulated into pellets in the wet state, according to the teachings of Patent No. 1,245,700, by William C. Geer, and it was made by mixing 400 parts of black with 200 parts of kerosene by weight in a Daisy churn, agitated for thirty minutes at 755 r.p.m., and the granules screened through a 14 mesh screen, and retained on a 60 mesh.

[fol. 172] Q. Was the product dried in any way to drive off the kerosene?

A. Yes.

Mr. Smith: The container which has just been identified by the witness, together with its contents, are offered in evidence as Defendant's Exhibit No. 27.

Mr. Edmonds: May I ask a question about it?

Mr. Smith: Yes.

Mr. Edmonds: Was that sample made recently and without any notice to the plaintiff and without any representative of the plaintiff being present?

Mr. Smith: Are you asking me or the witness?

Mr. Edmonds: Either.

Mr. Smith: It has been made recently and without notice to the plaintiff * * * [327-332].

* * * (Defendant's Exhibit No. 27, filed.)

By Mr. Smith:

Q. Dr. Drogin, I show you a chart, which I will ask to be marked for identification, Defendant's Exhibit No. 28, and ask you to look at that chart and state whether or not that is a diagrammatic representation of the method which you employed in your study of the Geer patent for the production of the sample which constitutes Defendant's Exhibit No. 27.

A. This chart represents the steps I took in preparing the sample of pellet black which I testified as representing sample No. 23 made by the Geer process.

Mr. Smith: The chart which has been referred to by the witness is now offered in evidence as Defendant's Exhibit No. 28.

(Defendant's Exhibit No. 28, filed.)

Q. Dr. Drogin, I now hand you a copy of United States Letters Patent No. 1,286,024, issued to Knowlton and Hoffman, and ask you if you have ever studied that patent.

A. Yes.

[fol. 173] Q. Will you please describe what you did in your study of this patent?

A. I prepared pellet black, friable grains and small lumps, according to the teachings of this patent.

Q. Now, will you please state what you did in making these studies, particularly with regard, first, to the proportions of black and water which you used, if you used water.

A. According to my data, I find that I used a mixture of black and water in the proportion of one to one and a half in one case——

Q. Which is the black and which is the water in those proportions, Dr. Drogin?

A. I used one part of black and one and a half parts of water.

Q. Did you use any other proportions of black to water in studying this patent?

A. No, as it was not necessary because it is not specified the exact proportions that should be used in order to make those friable grains. All that is indicated in the patent is to the effect that to mix enough of it to produce the desired condition.

Q. Now, what did you use in the way of mixers for studying the Knowlton and Hoffman patent?

A. I used, in one instance, a Daisy churn.

Q. And that is the same churn that you used for your other studies, as represented by Defendant's Exhibit No. 12?

A. Yes.

Q. Did you subject the mixture in the churn to different periods of agitation?

A. Yes.

Q. I hand you a container which is marked United Carbon Co., Inc., sample No. 3, and I will ask you to examine that and the contents of the container and tell us what it is.

A. Sample No. 3 is a pellet black made by granulation in the wet state according to the teachings of Patent No. 1,286,024 by Knowlton and Hoffman.

Q. Was that made in the Daisy churn?

A. Yes, sir.

Q. How long was it agitated?

A. That was agitated seven minutes and the sample that I see before me represents the pellet black screened [fol. 174] through a 14 mesh and retained on 20.

Mr. Smith: I will ask that the sample which has been referred to be received in evidence as Defendant's Exhibit No. 29.

(Defendant's Exhibit No. 29, filed.) • • • [333-336].

By Mr. Smith:

• • • Q. I now hand you a container which is marked with the tag of United Carbon Co., Inc., sample No. 9, and ask you if you can tell us what that is.

A. Sample No. 9 represents a sample of pellet black, made by granulation in the wet state, according to the teachings of Patent No. 1,286,024 by Knowlton and Hoffman.

Q. And what type of mixer did you use in making that sample, Dr. Drogin?

A. I used the Daisy churn.

Q. And how long was that sample agitated?

A. That was agitated for thirty minutes.

Q. And what did you find that particular sample represents as far as sizes of the particles is concerned?

A. This sample represents pellets and granules screened through a 14 mesh and retained on 20.

Mr. Smith: The sample which has just been identified by the witness is now offered in evidence as Defendant's Exhibit No. 30 • • • [336-337].

• • • (Defendant's Exhibit No. 30, filed.)

By Mr. Smith:

Q. I now hand you a container which has on it the tag of United Carbon Co., Inc., sample No. 18, and I ask you to examine this and tell us, if you can, what that is.

A. Sample No. 18 is pellet black produced by granulation in wet state, according to Patent No. 1,286,024 by Knowlton and Hoffman.

Q. What type of agitator or mixer did you use on that?

A. I used the Daisy churn.

[fol. 175] Q. And how long was that sample agitated and at what speed?

A. This material was produced by agitation for one hour at a speed of 168 r.p.m.

Q. What are the sizes of the particles represented in that sample?

A. They represent material screened through a 14 mesh and retained on 40.

Mr. Smith: The sample just identified by the witness is offered in evidence as Defendant's Exhibit No. 31 • • • [337-338].

• • • (Defendant's Exhibit No. 31, filed.)

By Mr. Smith:

Q. Now, in all of the samples, Defendant's Exhibits Nos. 29, 30 and 31, I believe you have stated that the proportion of black to water was one part of black to one and one-half parts of water; is that correct?

A. Yes.

Q. Now, in connection with the specimen, Exhibit No. 29, which is sample No. 3 of the United Carbon Co. label, can you give us the speed of agitation used in making this sample?

A. That speed was 755 r.p.m.

Q. Now, in connection with the sample, Defendant's Exhibit No. 30, United Carbon Co., Inc., tag, sample No. 9, will you please give us the speed you agitated that, or the speed of agitation used in making that sample?

A. That was 755 r.p.m.

Q. Now, in your studies of the Knowlton and Hoffman patent, did you use any other kind of mixer than the Daisy churn mixer, which you have just referred to?

A. Yes.

Q. And in using other types of mixers, did you vary the proportions of water to black? I refer you particularly to your sample No. 19 in connection with this question.

A. In the preparation of this sample I used one part of black to one part of water, ratio of 400 of black to 400 of water.

[fol. 176] Q. And what did you use as the mixer for that particular sample?

A. I used what is known as a Tamms mixer, a paint mixer.

Q. I will hand you a circular of the Tamms paint mixer and agitator, which has been marked for identification, Defendant's Exhibit No. 32, and will ask you to look at that picture of the apparatus, at the top of this circular, and state whether or not that is the Tamms mixer to which you have just referred.

A. Yes, it is.

Q. Can you describe briefly the operation of this mixer?

A. Principally the function of this mixer is to give the contents in the can a thorough shaking.

Q. And how is this accomplished mechanically, if you know?

A. The mixer is fixed on a base which is caused to rotate at a speed—to turn, rather, at a speed of around 335 r.p.m.,

the power supplied by a quarter horse power motor, and we get a shaking motion that is similar to the action I describe or indicate here now with my hand.

Q. Giving a vigorous rotary motion to the can which is secured on the holder?

A. Yes.

Q. Now, in making the sample, which is marked with the United Carbon tag No. 19, how long was the mixture of equal parts of black and water agitated or worked on the Tamms mixer?

A. The material was shaken for twenty-five minutes.

Q. And what does the sample specimen, which is in the container No. 19, represent in the way of sizes of particles?

A. It represents granules and pellets screened through a 14 mesh and retained on a 60.

Mr. Smith: At this time we will offer in evidence the circular the witness has referred to as Defendant's Exhibit No. 32.

(Defendant's Exhibit No. 32, filed.)

Mr. Smith: I will now offer in evidence as Defendant's Exhibit No. 33 the sample, No. 19, which has just been referred to by the witness.

[fol. 177] Mr. Edmonds: May the plaintiff's objection to all these recently made samples be noted on the record?

The Court: Let it be so noted that the plaintiff objects to testimony with regard to carbon black made by the witness for the purpose of this testimony, without notice, and not in the presence of plaintiff nor its counsel, and that the objection is overruled and an exception noted, and the objection shall be considered as applying to all such testimony by this witness; is that broad enough Mr. Edmonds?

Mr. Edmonds: Yes, sir.

(Defendant's Exhibit No. 33, filed.)

By Mr. Smith:

Q. I now ask you to refer to sample No. 15, which is Defendant's Exhibit No. 4, and will ask you to state how this sample was made.

A. Sample No. 15 was made by the granulation of flocculent carbon black in the wet state, according to the teach-

ings of W. C. Knowlton and H. H. Hoffman in their patent No. 1,286,024.

Q. And what type of mixer was used in making this sample?

A. I used a Pin drum.

Q. And what proportion of carbon black to water did you use?

A. I used the proportion of one part black to three parts water, using 900 grams of black and 2700 grams of water.

Q. Now, look at the chart which I have placed on the easel, and which is marked for identification Defendant's Exhibit No. 34, and state whether or not that represents diagrammatically the method which you employed in making sample No. 15, which is Defendant's Exhibit No. 4.

A. This chart represents the method I used in preparing the pellet and granule black which I have in sample marked No. 15.

Q. That sample No. 15 is Defendant's Exhibit No. 4, is it not?

A. Yes.

[fol. 178] Q. Now, how long was the material mixed in the pin drum mixer in making this sample?

A. This particular sample was obtained by rolling the pin drum for ten minutes.

Mr. Smith: The chart which the witness has just referred to is now offered in evidence as Defendant's Exhibit No. 34.

(Defendant's Exhibit No. 34, filed.)

Q. I will ask you now to look at the Trent British Patent No. 183,430, and ask you if you have made any studies of this patent.

A. Yes.

Q. Will you please outline briefly what you did in studying this patent?

A. Yes, sir.

Q. First, you might tell us what materials you used.

A. I used a bituminous coal which had been pulverized to pass a 200 mesh screen.

Q. And was that coal mixed with any kind of wetting liquid?

A. Yes.

Q. Please state what that liquid was.

A. I mixed that with water until I got it thoroughly wet.

Q. How was this mixing done?

A. That was done in a Daisy churn.

Q. Similar to the churn, Defendant's Exhibit No. 12?

A. Yes.

Q. And how fast were the paddles revolved in this mixing?

A. Well, the agitation was done by hand, I mean the paddles were revolved, or this machine was operated as it stands there, by turning the crank, by hand.

Q. So that it would be the speed of the paddles depending upon the strength of the operator?

A. Yes.

Q. Now, after you had mixed the coal with water in the Daisy churn, what was done then?

A. Then I added kerosene.

[fol. 179] Q. Approximately how much kerosene was added?

A. I added 770 parts of kerosene.

Q. Do you have a record of how much coal and how much water was used in making this test?

A. Yes, I used 550 parts of finely divided coal with 4,400 parts of water and then added 770 parts of kerosene.

Q. Now, after the kerosene was added, what was done then?

A. I then agitated it until the coal was agglomerated and came to the top.

Q. And what was the nature of those agglomerates?

A. They were pellets and granules of different sizes.

Q. I now hand you a container which is marked with United Carbon Co.'s tag as No. 1, and ask you if you will please state what this container contains.

A. This container contains pellets of coal made by the flotation process, prepared according to patent 183,430, granted in Great Britain, patent issued to the Trent Process Corporation, and it was made by mixing 550 parts of finally divided bituminous coal with 4400 parts of water, agitating same until the coal was thoroughly wetted and then adding 770 parts kerosene, and agitating same until the coal was agglomerated into substantially spherical agglomerates, and after drying these agglomerates they were screened through a 14 mesh and retained on 60.

Mr. Smith: The specimen which has just been identified by the witness is now offered in evidence as Defendant's Exhibit No. 35.

(Defendant's Exhibit No. 35, filed.)

Q. I believe that I neglected to ask you, in connection with the specimens which you have identified as having been made during your studies of the Knowlton and Hoffman patents, particularly Defendant's Exhibits Nos. 29, 30, 31 and 33, whether or not those pellets were dried after they had been formed in the particular mixers which you used.

A. They certainly were dried.

Q. And in each instance the water was evaporated from the pellets?

A. Oh, yes.

[fol. 180] Q. In connection with your work in the carbon black fields, Dr. Drogin, particularly the manufacture of dustless carbon black, do you know whether there is any minimum bulk figure, or minimum density of the dustless products which is generally considered to be the lightest that these materials can be made and still be successfully handled in the bulk handling systems?

A. Yes.

Q. Can you state what this density is?

A. It is held that a pellet black which bulks less than 20 pounds to the cubic foot is not quite suitable for commercial use or for commerce, in view that it tends to produce much more dust than is desirable or advisable.

Q. And what is the dust due to?

A. The dust is due to the disintegration of the pellets, the disintegration taking place during transit and handling of the material • • • [338-346].

• • • Cross-examination • • • [347].

• • • Q. Dr. Drogin, you are the author of this article, a portion of which appears in Plaintiff's Exhibit No. 1; are you not?

A. Yes.

Q. That was published in a trade magazine in September, 1935; was it not?

A. Yes.

Q. And what is stated in there represents what you knew or thought at the time, and what you still think?

A. Yes.

Q. That is right, is it not?

A. Yes * * * [347].

* * * (Plaintiff's Exhibit No. 23, filed.)

Q. This other magazine article in Plaintiff's Exhibit No. 1 you recognize, do you not: "History, Use and Manufacture of Carbon Black" by Messrs. Allen, Price and Randall.

A. May I glance at this for a moment, please?

Q. Yes (handing paper to witness).

A. I recognize the name but I don't recognize offhand the article.

[fol. 181] Q. Messrs. Price and Randall were associates of yours in this work; were they not?

A. Yes.

Q. And they were well informed on the matter of carbon black, were they not?

A. Mr. Price was * * * [347-348].

By Mr. Edmonds:

* * * Q. Do you know what are the requirements of the rubber companies with respect to the purity of the pelleted carbon black that they buy from the carbon companies for use in their rubber?

A. Judging from this specification of the various rubber companies, they have certain standards as to what the purity of the carbon black should be.

Q. They are very specific, are they not?

A. At times, yes.

Q. Can you tell us just what those purity requirements are?

A. Primarily it is a question of grit, the amount retained on a 325 mesh screen.

Q. Do you know what the requirements are?

A. Some companies specify less than a tenth of a per cent. to be retained on this particular mesh screen.

Q. Is that the only requirement?

A. No, not the only one.

Q. All right, tell us the others.

A. Another requirement is moisture content.

Q. What is that?

A. That is the percentage of moisture retained in the carbon black after——

Q. What is the requirement; what does the rubber company specify?

A. It depends on whether they receive the material in bags or in tank cars. In bags it would be slightly more than two per cent., or sometimes under three per cent., or in tank cars it is one per cent. or under.

Q. Now, the requirement as to the purity of the pelleted black, is there anything else?

A. The ash content is another requirement.

[fol. 182] Q. What is that ash exactly, if you can state it?

A. I believe that some companies specify the black should not show in excess of a half of a per cent. of ash.

Q. Do you have any specific knowledge on that point? We are trying to get it very definitely now.

A. Yes, my knowledge is——

Q. All right; tell us the specific requirement.

A. You see, all companies do not have the same requirement. Some don't even mention it, where others do, and those who do, are not all agreed on the exact percentage that should be retained.

Q. All right. Tell us what the requirement is as exactly as you can fix it, and you can give the company if you want.

A. Not having seen an actual specification for quite a while, I can only quote from memory, but it is my belief that in one case, I think it is a half of one per cent. maximum ash.

Q. Is there any other item entering into the rubber companies' specification of purity?

A. I believe there is another one called acetone extracts.

Q. Tell us what you know about that.

A. Those that do specify it, I believe, have a figure of three per cent. maximum.

By the Court:

Q. Of what?

A. I mean three-tenths of a per cent. acetone extract. In other words, when the black is heated under reflux condenser with acetone, the acetone should not take out more than three-tenths of a per cent., nor the residue acetone, when

evaporated by drying, should not show that it has taken out more than three-tenths per cent.

By Mr. Edmonds:

Q. So that, generally speaking, these requirements as to purity, are rather rigid, are they not?

A. To some extent; to a certain extent, yes.

Q. And the United Carbon fulfills those requirements as to purity, does it not?

A. It certainly does; otherwise it would not sell it.

[fol. 183] Q. You said the density of the Sayre product is about 25 pounds—25.4 to be exact.

A. Yes.

Q. Now, this Wiegand and Venuto patent in suit makes a recommendation, does it not, as to the density which a product might well be?

A. I did not quite understand that.

Q. Don't you recall that in the specification of the Wiegand and Venuto patent it is stated that it would be well to make the density of the pelleted product so and so?

A. Yes, it says—it refers something to having a satisfactory density.

Q. Is that all it says, "satisfactory"?

A. If you will show me the patent——

Q. Don't you have a copy of the patent at hand?

A. No.

Q. All right, look at page 2, lines 94 to 98 of the patent.

A. Yes.

Q. There the recommendation is that the pellets be of a density about double that of uncompressed black; isn't that right?

A. That is right.

Q. And that is just what the Sayre pellets are?

A. Practically so.

Q. Now, this Wiegand and Venuto patent also gives you adequate directions for procedure whereby you could make the pellets of the size that you would like to have them be; does it not?

A. It indicates directions how to make certain black by the flotation process.

Q. But that is not what I asked you. I asked you if the specifications give you directions as to how to control the

size of the pellets so that the final pelleted product will be of that size which you would like to have.

A. It does recommend directions how to control the size, but it doesn't specify the exact size.

Q. No; but it gives you directions which you can follow.

A. Yes.

[fol. 184] Q. And arrive at that size which you, yourself, select?

A. It gives directions how to control the sizes, but it does not tell me whether those directions will give me a size of such specific dimensions.

Q. Very true. Now, in accordance with those directions there as to how to proceed to control the size, could you operate that process in such way as to produce pellets of the same size as the Sayre pellets?

A. I believe that a working out—that is, by trial and error method, by repeated trials, you could arrive at the conditions which would give a pellet the size of the Sayre.

Q. Naturally, because you have said that the directions for controlling the size are there. Now, it also recommends that the pellets be of about double the density of uncompressed black, namely, about 25 pounds to the cubic foot, so I take it it follows from what you have said that one could, by following the directions of the patent, make pellets which are of the size of the Sayre pellets, and which are of the density of double the density of uncompressed black?

A. Yes.

Q. Then those pellets made by this Wiegand and Venuto procedure would be pellets which are the same as the Sayre pellet in size and the same as the Sayre pellets in density, or rather in weight per cubic foot?

A. Yes.

Q. Now, would those pellets be as porous as the Sayre pellets?

A. Much more porous.

Q. We are talking about two pellets now. Let us get down to two pellets: one the Sayre pellet and one the Wiegand and Venuto; and they are of the same size and of the same weight, will one be more porous than the other? If you are thinking about the condition of the interior, we will come to that later. At the moment I am asking you about the porosity, the over-all porosity. Will one of them be more porous than the other?

A. In answer to this question, you assume that not only they are of the same size but of the same weight.

[fol. 185] Q. We came to that by the——

A. (Interposing) And in my experiments I was not successful in getting a pellet that was the size of the—that is, I was not successful in making a Wiegand and Venuto pellet according to the patent, of the same size and yet of the same weight.

Q. Now, you will recall that I have not yet asked you anything about anything that you named. I asked you about the directions in the patent, and we agreed that those directions are adequate to tell you how to make a pellet of a size that you select, and therefore we agreed that one could make a pellet of the size of the Sayre pellet, and of the weight of the Sayre pellet, so I asked you are those two pellets, the same as to size and weight, of the same porosity or of different porosity.

A. A pellet that is made by the Wiegand and Venuto process is very porous within, and even though its size may be the same as that of a Sayre pellet, I cannot offhand say that—well, in fact, they cannot be of the same weight, judging from my own experience.

Q. Now, I will ask you to go back a minute to the question where we agreed that these two pellets that we are talking about are of the same weight, so it gets you no where for you to say one is of different weight from the other.

A. I beg your pardon; I did not say they were both the same weight.

Q. Well, then, we will have to start at the beginning. You said that the patent has in it directions for making a pellet of a size you select.

A. That is correct.

Q. So I asked you to consider a pellet made of the size of a Sayre pellet.

A. That is right.

Q. And like the Sayre pellet in weight, both of them of a weight represented by 25 pounds to the cubic foot. It is now those two pellets, the same in size and weight, about which I ask you with respect to porosity. Are they the same in porosity or different?

A. May I reiterate that, according to the general direc-

[fol. 186] tions in the patent, the statement is made that cer-

tain conditions must be varied in order to influence the size. There is no specific statement made as to the actual size of it, or as to the definite dimensions of the pellet. Now, I stated that it is possible, by trial and investigating of the conditions, to produce a pellet by the Wiegand and Venuto process which would be of the same size as that produced in the Sayre plant. Now, I also agreed that the patentee says that bulk of the material, according to his statement, should be approximately twice the number of pounds of the uncompressed black, but I cannot agree, judging from my own experience and experiments, that when making a pellet from the Wiegand and Venuto process, it would meet the two conditions not only of the same size of the Sayre pellet but also of the same weight. I have not been able to produce a pellet by the Wiegand and Venuto process that would bulk 25 pounds to the cubic foot.

Q. Have you finished?

A. Yes.

Q. Can you consider two pellets, one a Sayre pellet, and the other a Wiegand and Venuto pellet, the same in size and the same in weight, the weight being that represented by 25 pounds to the cubic foot—will both of those pellets have voids within their interiors with air in those voids?

A. The Wiegand and Venuto pellet will have considerable voids; the Sayre pellet hardly any.

Q. Let me ask you a very simple question about the patent and the processes. If you take two pellets of the same substance, of the same size and of the same weight, will not of necessity the degree of porosity be the same?

A. The total degree of porosity will be the same.

Q. Do not you want to amend your last answer you gave, which is certainly not in harmony with what you said to his Honor?

A. Mr. Edmonds, you have assumed they are both of the same size and the same weight. I told you honestly from my experience that I might get them of the same size but I could not get them of the same weight.

[fol. 187] Q. But I have not asked you anything about whether you could make them of the same weight or not. For the purpose of this question I ask you to consider two pellets, whether you made them or someone else, they are the same size, they are the same weight and they are

substantially pure carbon black. Their weight is that represented by 25 pounds per cubic foot. Now I ask you if the total voids in one will be the same as the total voids in the other?

A. Yes.

Q. And if one is porous the other is porous? There is no difference between them in total of porosity, is there?

A. That is right.

Q. If I have understood you, you think there is some difference between those two as to the nature of that porosity, that difference being that in the Wiegand-Venuto pellets the voids are larger and of less number whereas in the Sayre pellets they are smaller and of greater number, is that an accurate statement of what you say?

A. No.

Q. Then I must have misunderstood you, so I will ask you to try to be more specific in defining what you say is the difference between these two pellets of the same size and of the same weight, both being porous and being porous to the same degree: what is the difference between them as to the nature of that porosity?

A. In the case of the Wiegand-Venuto pellet you would have a spongy interior, porous, but the hardness of the shell would compensate what the pellet lost in density in the interior of the shell; whereas in the Sayre pellet you find a much more uniform interior, perhaps one single or two voids, and the shell not extending out so markedly that one would construe that the outside or exterior portion was making up for something that was lacking within.

Q. Then is this an accurate statement of your views: that in the Sayre pellet the porosity is distributed rather uniformly throughout the mass, whereas in the Wiegand-Venuto pellet that uniformity is not so marked, particu- [fol. 188] larly in that there is greater porosity near the center and much less at the exterior, where there is more or less of a shell? Is that a correct statement of your view?

A. No, I would not say that the porosity is uniformly distributed.

Q. More uniformly distributed, I said, with the Sayre pellet.

A. If you consider the nature of two voids in a pellet surrounded by dense material and extending from there

that void to the outside part of the pellet, if you consider that uniform, yes. I would not.

Q. Do you think of a Sayre pellet as one which has a couple of voids in near the center and no other voids of any size anywhere else in its makeup?

A. No, I have seen certain pellets that had no void, they were just solid.

Q. How much did they weigh per cubic foot?

A. I did not weigh it.

Q. How much would it have to weigh if it were pure carbon?

A. According to our process we find it will produce somewhere between 25 and 27 pounds per cubic foot.

Q. Do you think that this pellet of pure carbon, having no voids in it at all, would weigh 25 pounds per cubic foot?

A. When I say "no voids" I mean the voids that are perceptible in these microscopic examinations. The Sayre pellets do have some voids, indicating the fact that they drink up some liquid when immersed in it.

Q. And the total of the voids is the same as the total voids in any other pellet of the same size and of the same density?

A. If you have such two pellets I would say yes.

Q. Going back for a minute to this matter of porosity, does the rubber manufacturer have any specifications of the degree of porosity that you know of?

A. No.

Q. So far as you know, is he aware of the porosity of the pellets you sell him beyond knowing what they weigh per cubic foot?

A. Not that I know of.

[fol. 189] Q. Do you know of anything in rubber manufacture that is dependent in any way upon the degree of porosity of the pellets of carbon black?

A. Not that I know of.

Q. So far as you know, the rubber manufacturers who have been buying these pellets to use for years are totally unaware of any differences there might be in the kind of porosity within the pellet that they buy?

A. I never discussed that subject with them.

Q. And the quality of the rubber is not influenced in the least by the nature of the porosity of the pellets, is it?

A. The quality of the rubber?

Q. Yes, made with carbon black pellets. That is not changed at all by reason of the difference in the degree of porosity of the pellet, is it?

A. Not that I know of.

Q. The essential thing is that they be pellets of substantially pure carbon black and that in the process they break down to the individual particles, that is true, is it not?

A. That is generally true.

Q. Now, you made some pellets in accordance with some of your ideas about this Wiegand-Venuto patent. I want to ask you one or two questions about that. Yours was a batch operation, was it not?

A. Oh, yes.

Q. One suggestion that the patent makes with respect to making these pellets in a batch operation is that you employ violent shaking. You did not do that, in your operation, did you? I am asking about your operation, —did you do that with your operation?

A. I shook it and agitated it.

Q. I am sure you agitated it because you explained that. I asked you whether you employed a violent shaking?

A. Yes, I employed violent shaking in some of the batches I prepared in accordance with this process.

Q. Let us talk about the one you detailed here and of which you gave us a sample of your product. In that batch operation did you subject the mixture to violent shaking?

A. No.

[fol. 190] Q. Did you subject it to agitation by rotating or tumbling the container?

A. No, I did not rotate or tumble the container.

Q. This patent also says, as we have agreed, that it would be well to make the pelleted product of a density twice that of the uncompressed black: you recall that?

A. That is what the patent would like to do.

Q. And this product which you made was not of that density, was it?

A. It was a density of about 17 pounds.

Q. In other words, you raised the density of the carbon black only half way as far as the patent recommended?

A. Maybe that was wishful thinking of the patent.

Q. Yes, maybe it was wishful thinking, Mr. Drogin, of yourself too; but the fact is you raised the density half way to what the patent stated?

A. That simply followed the procedure I used in preparing a particular batch.

Q. We are agreed on the fact that you raised the density half way?

A. I raised the density to about 17 pounds per cubic foot.

Q. From 12½ pounds, approximately?

A. Yes, about 12 or 13 pounds.

Q. And the patent suggests you raise it from 12½ to 25 pounds?

A. That is right.

Q. You went half way?

A. That is right.

Q. You went that half way by using some apparatus other than what the patent recommends?

A. I do not know whether it recommends a definite apparatus.

Q. But you just agreed that you did not use violent shaking or rotating or tumbling of the container. So, according to that, I should think you would have to agree you did not use what the patent recommends.

A. The purpose of violent shaking and rotating the container is to produce the agitation of the mass within.

[fol. 191] Q. I am going to give you the procedure you actually used. At the moment are we not agreed that you did not use the apparatus recommended in the patent and did not arrive at the density recommended in the patent?

A. According to your line of questioning, I would say yes.

Q. At one point in the patent the recommendation is that the operation be continued for five minutes.

A. That is right.

Q. That you did follow?

A. I did.

Q. That and that only?

A. And the proportions of black and water and gasoline.

Q. So you followed the time and the proportions but did not use the apparatus?

(No response by the witness.)

Q. We will pass that for a minute if you do not care to answer. Let me ask you this—as to the agitation of that mixture, was the agitation it got in a churn like the one shown here?

A. That is right.

Q. Suppose that churn had been exactly what it was except that the jar was twice as big, the agitation would have been much less, would it not?

A. I presume so.

Q. You do not presume it, you know it.

A. I did not try it.

Q. Some things you do not have to try. You know that agitation would be much less, do you not?

A. I could not say offhand until I actually saw it in a big jar.

Q. Suppose that jar had been somewhat smaller, we will say, an inch smaller both ways, then the agitation would have been much more, would it not?

A. I do not know, I did not try that.

Q. Maybe someone might think it was rather serious for you to say you did not know that. I would think you would say that is a thing you would know without [fol. 192] trying. Do not you want to reconsider those answers?

A. No. I did not try those things so I cannot truthfully tell you whether or not there would be a difference in agitation.

Q. I ask you this. Suppose all the conditions of operation which you performed remain unchanged except that the size of the jar of that churn were varied up and down from what it actually is. Then the agitation of the material would be subject to variation with those changes of dimensions, would it not?

A. I said, I presumed so.

Q. And therefore the actual amount of agitation of the product which you made was the result of the selection made by the designer of that churn as to how big he would make his churn—does not that follow?

A. It followed I made the pellets by using this churn I used in my experiment.

Q. And the degree of agitation was determined by the selection by the churn designer of the dimensions used; that is true, is it not?

A. It is true that you get that. You could certainly get a certain degree of agitation by the power or the quantity of the rotation of the panels themselves.

Q. But that is not what I asked you. I specifically said everything was the same except the dimensions of that bowl. You would get different products, would you not, using bowls of different sizes, all other factors remaining the same?

A. I cannot quite subscribe to this. You would get different Wiegand-Venuto pellets.

Q. Different sizes?

A. You would get Wiegand-Venuto pellets differing in size dependent on the agitation.

Q. Using a churn, that would depend on the size of the bowl if everything else remained the same, would it not?

A. I presume so.

[fol. 193] Q. Now, in speaking of the product of this operation of yours in making what you call Wiegand-Venuto pellets—in speaking of these Wiegand-Venuto pellets which you made you stated they were better looking than the pellets which the plaintiff produced here as pellets made by the Wiegand-Venuto procedure. That is your testimony, that they are better looking?

A. I do not remember what you mean. Read it to me, please.

Q. On page 139 the question is, "Now, so far as the separation and the flotation of the pellets was concerned, did you secure much the same sort of separation and flotation that was secured in the demonstration apparatus here yesterday?" Your answer was, "To a degree, yes, but much better looking." Is that your testimony, that those pellets you produced as made by you are much better looking pellets than the ones made in court here by the plaintiff in accordance with the Wiegand-Venuto process?

A. I mean to imply by that simply that in the experiment that was tried here it was my impression that the end point had not been quite reached. By the "end point" I mean getting a pellet that looked rather spherical and of smooth surface, the same as described in the patent. That was my impression when I looked into the cylinder at that time.

Q. Do you mean to testify that those pellets that you produced as having been made by you by the Wiegand-

Venuto procedure were more perfect in end point, or whatever it is that you referred to by that name?

A. I did not mean to say they were more perfect. That is the procedure I followed in the laboratory for making these and they gave me a product that gave a sharper end point and the pellet looked more like what we tried to duplicate according to the patent.

Q. I will ask you to look at a couple of samples. Which of those two would you say is the better looking product?

A. Those on this end are rather large and these are very small. Ordinarily seeing them they are too small at first [fol. 194] glance and the difference in size is such that I would have to have larger magnification.

Q. Which would you call the better looking product?

A. It is hard to say. They both look good but some look better than others. One looks better than the other. It is hard to say, Mr. Edmonds, the question of which is better than the other.

Q. This being the one on the left?

A. You can see a few pellets that are round, but the same thing might be said of some of the big ones in here.

Q. Refer to the one on the right.

A. I believe in my testimony the expression "better looking," I think you will find that the production was to a better degree consummated in the case of our procedure than according to yours.

Q. Do you have any views on the subject of which of these pelleted blacks would be more acceptable to the rubber manufacturer?

A. That is hard to say because, in a sense, from my experience, both types would be acceptable to the rubber industry as far as appearances goes.

Q. Assuming both of them had the quality of a commercially pure carbon, both would be accepted by a rubber manufacturer who wanted pelleted carbon black?

A. Yes.

Q. Do you think that is flowable without breaking down to considerable dust?

A. That I cannot say.

Q. The one on the left quite obviously is pelleted?

A. Possibly, so is this one.

Q. You think they both would flow?

A. Yes; and as far as breaking down with dust, I am not prepared at this instant to tell you which is which.

Q. Are you still unwilling to say which is the better looking? You used that expression in your direct examination?

A. You might have misunderstood my expression—"better looking." I was not thinking in terms of beauty.

[fol. 195] Q. You used the expression. I want you to explain what you mean as applied to these two samples.

—. I did explain that my expression of "better looking" referred only to voids in the cylinder when it was made.

Q. It had no reference to utility in rubber manufacture?

A. No.

Q. And it had no reference to flowability?

A. No.

Q. Nor the condition of hardness?

A. No, I did not examine it for that.

Mr. Edmonds: Let the reporter please note on the record that the carbon black in the tray which I hold in my right hand is carbon black from the sample of black made by this witness in accordance with his notion of the procedure in the Wiegand-Venuto patent. The specimen that I held in my left hand during this examination of the witness is carbon black from the lot recently made up in accordance with the direction in the Wiegand-Venuto patent; this being taken from Plaintiff's Exhibit No. 11 * * * [349-372].

* * * Q. You said, if I remember right, that the rubber companies would not accept pelleted black if it were of a density of 20 pounds to the cubic foot or less, did you not?

A. I did not say they would not accept it.

Q. What was your statement about that?

A. I believe I expressed an opinion that it was not a desirable product in view of the greater amount of dust caused by the breaking down of the pellets in transit and in handling.

Q. So the pellets that you made by the Weigand-Venuto procedure were made in an undesirable form, were they not?

A. I would not say they were in an undesirable form. I simply say they were made—I did not send them to the rubber company.

Q. Would you say it is undesirable from the rubber company's point of view to have a pellet black of a density less than 20 pounds to the cubic foot?

A. That is right.

[fol. 196] Q. And you made those Wiegand-Venuto pellets of less than 20 pounds. Mr. Hanson made some suggestion that the procedure of this Wiegand-Venuto patent could not be used very well in a continuous process. Do you hold that view?

A. I believe he said something about a continuous process. My recollections were that it could be made continuous if you went to considerable trouble. That is my recollection of his testimony.

Q. You agree to that—it may be continuous or batch, as you please?

A. I believe it could be continuous.

Q. When you were making some samples in accordance with the Knowlton patent of which you spoke, did you put in starch in the mixture?

A. No, sir.

Q. Did you put in glue or dextrose or sugar?

A. No, sir, I used nothing but water and black.

Q. That seems to be what he recommends, namely, to put in starch or glue or dextrose or sugar?

A. May I look at the patent, please?

Q. He gives a specific example, does he not?

A. He also stated you could mix black and water in suitable proportions and obtain concretion of the black into friable grains.

Q. But he does give you a specific example of what he proposes, does he not?

A. May I look at that? He gives you a specific example—he says on page——

Q. Won't you answer the question—he does give you a specific example?

A. Yes, he does cite an example.

Q. And that example includes a certain number of parts of starch?

A. That does.

Q. But you did not do that?

A. No.

Q. And such starch would be a binder of particles in agglomerate form?

A. According to Mr. Knowlton and Hoffman in their example.

Q. Look at this chart behind you that you have prepared in connection with the Knowlton and Hoffman patent, De- [fol. 197] fendant's Exhibit 34. Have you made any of that here in court?

A. I do not know. These were taken from a batch that was prepared. Since we had a small container we filled it up to about two-thirds and called it "quits."

Q. That product in that little vial or container at the extreme right-hand corner of that exhibit is distinctly different from the product which has been offered here as Exhibit No. 4?

A. When you say distinctly different, what do you mean to imply, Mr. Edmonds?

Q. There is quite a decided difference in the size of the pellets, is there not?

A. Yes, I would say those in the bottle are a little bit larger.

Q. That is Exhibit No. 4?

A. It is a little bit larger.

Q. Larger than those on the chart?

A. Yes.

Q. Is there any other difference visible?

A. Not that I could tell by looking at it offhand.

Q. As I understand it, in making this product you put carbon black through this pin drum?

A. That is right.

Q. Was there a shaft going through the drum that had pins on it?

A. No, there was no shaft.

Q. In this sample there was none at all?

A. No.

Q. How fast did the drum rotate?

A. 58 r.*m.p.*

Q. 58 r.p.m.?

A. That is right.

Q. How many pins were in it?

A. If you wish I can show you the pin drum itself. I do not recall the exact number.

Q. Have you the actual drum in court that you used?

A. Yes (exhibiting a drum).

Q. You were asked a few questions about the Geer patent and about the procedure adopted which you thought of as in accordance with that patent. Among other things you said you dried the product to drive off the kerosene. [fol. 198] That is exactly contrary to what Geer says, is it not?

A. Not contrary.

Q. Is it not contrary to him and did not he say that some of the kerosene evaporated on the rolls and that the balance evaporated in the finished rubber? Do not you find that specifically stated in the patent?

A. Yes, it is mentioned in the patent.

Q. So what you did here was contrary to what the patent stated,—that follows from what you stated.

A. What I did was to obtain granulated black.

Q. You are answering something not asked you. The fact is that what you did is contrary to the statement in the patent, is it not?

A. Yes.

Q. You mentioned the Trent British patent. Do you know when that churn mentioned came on the market?

A. I do not recall.

Q. Do you know when that paint mixer came on the market?

A. I do not recall.

Q. Going back to Trent, those Trent patents state over and over again, do they not, that the final product is an agglomerate of coal and oil?

A. I believe, according to the patent, you are supposed to agglomerate the coal, and, naturally, as it is formed, it would contain coal and oil.

Q. Does it so state in the patent time after time?

A. I do not know about "time after time."

Q. As a matter of fact, do not you know just from your reading that for 100 years or more people have been making briquets out of fuel and a binder such as oil. They used anything that will burn, like peat, lignite or low grade coal, or even high grade coal, and those briquets have been made for generations back of such combustible material and oil,—generally low grade oil?

A. Yes.

Q. And a very common procedure over the past 100 years has been to mix up quantities of fuel and oil and [fol. 199] put it in a press and press into a briquet; that is true, is it not?

A. That is true.

Q. What Trent did was to make those briquets by a different process, not requiring the use of such a process?

A. Trent did more than that. He eliminated the impurities of the coal by the flotation process, first mixing it with water and then adding this oil.

Q. And he eliminated that operation of pressing the briquets into a solid form?

A. I presume he did.

Q. But that was the same old briquet, a mixture of coal and oil?

A. He finished up with an agglomerate containing coal and oil * * * [373-379].

* * * Redirect Examination * * * [380].

* * * Q. In connection with the rubber companies' specifications as to grit, is there any difference between their specifications on what you would call loose black and the dustless black?

A. No, sir.

Q. Is the rubber company specification shown any different for loose black than for the pelleted black?

A. No.

Q. In connection with the acetone extract, is there any difference between the loose black and the dustless black?

A. No.

Mr. Smith: At this time we would like to offer in evidence as Defendant's Exhibit No. 36 the pin drum which was identified by the witness and marked for identification "Defendant's Exhibit No. 36."

(Defendant's Exhibit No. 36, filed.)

Q. Look at the chart which I just had placed on the easel which is marked for identification "Defendant's Exhibit No. 37." State whether or not that is an accurate diagrammatic chart of the experiments you performed in connection with the Wiegand-Venuto patent * * * [380].

* * * A. It is.

[fol. 200] Mr. Smith: I would like to offer the chart the witness has just identified as "Defendants Exhibit No. 37."

(Defendant's Exhibit No. 37, filed.)

Mr. Smith: I believe we have neglected to offer in evidence the jars which have been marked for identification as Defendant's Exhibits 10 and 11 that this witness referred to in his direct examination. I would like to offer them as Defendant's Exhibits 10 and 11 at this time.

The Court: Very well.

(Defendant's Exhibits Nos. 10 and 11, filed.)

Recross-examination • • • [381].

• • • Q. In your practice of the Wiegand-Venuto procedure you used 80 parts of water, and this chart says 40 to 80 parts?

A. I used 80.

Q. This chart specifies as to the gasoline 7 to 9 parts, and you used 9?

A. That is right.

Q. In other words you used the top figure of both water and gasoline?

A. I used one of the figures specified by the patentee.

Q. The patentee specified two ranges.

A. The limits.

Q. You took the top figure in each of the two ranges?

A. Yes, sir.

Mr. Smith: We have produced the figures regarding the dustless pellet shipments by the extrusion process which Mr. Hanson was asked to produce in his testimony by Mr. Edmonds. With the consent of Mr. Edmonds I will read those figures into the record at this time with the understanding that Mr. Hanson will be available for cross examination. Here are the figures that Mr. Edmonds wished for defendant's shipments of dustless pellet made by this extrusion process, the Sayre process and the Cabot process • • • [381-382].

• • • The Court: If you so desire.

[fol. 201] Extrusion Process

Dustless Pellet Shipments

Year	Amount Shipped
1934	2,000#
1935	84,875
1936	7,031,000
1937	26,205,000
1938	29,858,000
1939	48,578,000
1940	41,756,000
Total	153,514,875#

Dustless Pellet Shipments—Wet Process—Sayre Process—Drum Mixers

	Stinneti	Sayre	McIlroy	Sanford A	Total
1937	.....	.....	680,363	.....	680,363
1938	.....	.....	3,747,538	.....	3,747,538
1939	2,000	10,545,625	6,239,176	965,638	17,752,439
1940	4,983,313	12,893,460	5,858,400	6,273,280	30,008,453
	4,985,313	23,439,085	16,525,477	7,238,918	52,188,793

Dustless Pellet Shipments—Cabot Process

	Rock Creek	Combined Plant
1934	1,130,125	—o—
1935	16,037,378	3,656,294
1936	22,599,827	18,135,756
1937	18,348,078	11,928,742
1938	19,254,925	11,298,887
1939	19,869,162	11,533,200
1940	17,909,747	10,679,050
	115,149,242	67,231,929

* * * [383-384].

[fol. 202] Plaintiff's Rebuttal

Testimony of H. M. Parsekian

* * * Direct Examination * * * [385].

* * * Q. Will you give your name and age, please? A. H. M. Parsekian, 28.

Q. Your residence?
A. New York City.
Q. You are in the employ of Binney and Smith?
A. That is right.
Q. In what capacity?
A. I am at the head of their New York laboratory.
Q. How long have you been at the head of that laboratory of Binney and Smith?
A. About four years.
Q. Were you in the employ of the company prior to that?
A. Yes, sir.
Q. Working in the laboratory?
A. That is right.
Q. And your work in the laboratory, is that largely or entirely devoted to carbon black?
A. It is practically and entirely devoted to carbon black.
Q. That has been true practically all the time that you have been there?
A. That is right.
Q. I will ask you to look at a small can containing pelleted carbon black and state if you know what it is.
A. Yes I do.
Q. What is the carbon black contained in that can?
A. That is a sample of commercial production of pellet black made by the Wiegand-Venuto process which I received from the plant in Monroe, La.
Q. By the "Wiegand-Venuto Process" you refer to the two-liquid process described in the patent in the suit?
A. That is right.
[fol. 203] Q. When did that come into your possession?
A. According to the date on this can, in October, 1935.
Q. Independently of what is on the can and just as a matter of your recollection, is that the date when it came to you?
A. That is the date.
Q. Has it been in your possession ever since then?
A. Yes, sir.
Q. Right in the laboratory of the Binney and Smith Company?
A. That is right.
Q. And you received it from where?
A. From Monroe, La.

Q. At the plant where those pellets were made by the two liquid process when they were made commercially?

A. That is right.

Q. To the best of your knowledge and belief those are pellets made by that particular process and samples as made of commercial black in 1930 and 1931?

A. That is right.

Q. Do you know whether there is any other sample of that process in existence anywhere else in the world?

A. No, sir, I do not.

Q. Is that all you have?

A. I have a small vial taken out of there.

Q. During these last few days?

A. Yes.

Q. Will you tell us what is the density of the pelleted black in that can?

A. Very nearly 25 pounds per cubic foot.

Q. Very nearly,—that is, having a small fraction of a pound of difference?

A. Yes.

Q. Now will you look at this sheet of photo-micrographs that I hand you and state, if you know, what those pictures are and who made them?

A. I took those pictures with my assistants. They show the commercial two-liquid pellets you just referred to. [fol. 204] They show our present standard micronex beads, and they show the United Sayre beads.

Q. Just what were the particular two-liquid beads shown on the photograph at the top?

A. They were that sample.

Q. Exhibit 22? Those are from this can which you have just identified?

A. That is right.

Q. That is, the 1930 production of two-liquid beads?

A. Yes.

Mr. Edmonds: I will now offer in evidence this can which the witness previously referred to and which was identified, to be marked "Plaintiff's Exhibit No. 22".

Mr. Edmonds: I will offer this sheet that the witness has identified and ask to have it marked "Plaintiff's Exhibit No. 24."

(Plaintiff's Exhibits Nos. 22 and 24, filed.)

Q. Getting back to this Exhibit No. 24, the two figures at the top are the 1930 beads,—what about the next two?

A. The next two are beads representing our present production.

Q. This is typical present-day production of micronex beads as made by the plaintiff?

A. That is right.

Q. And the four photographs, are they all beads from Exhibit No. 20?

A. Yes, sir, they are.

The Court: What is Exhibit 20?

Mr. Edmonds: The defendant's product.

The Court: From what source was that derived?

Mr. Edmonds: It was offered to us by defendants as typical of the Sayre manufacture • • • [385-389].

• • • Cross-examination • • • [389]

• • • Q. In connection with the sample, Plaintiff's Exhibit No. 22, the sample in the tin can, do you of your [fol. 205] own knowledge know how these beads were made in this can?

A. I was told they were made by the two-liquid process.

Q. Who told you that?

A. I do not remember that.

Q. Is that all the knowledge you have as to the contents of that can and how it was made?

A. That is all I remember. We have records which will show.

Q. In connection with this sheet of pictures here, "Plaintiff's Exhibit No. 24", will you please tell us how the selection of the pellets in the paper was made, that is, how the two-liquid process picture was made, for the purpose of this photograph?

A. How it was cut?

Q. Yes.

A. We merely took that out of the can and put them in a cavity and took a picture of them.

Q. How was the fracture of the pellets there selected?

A. The fractures show what we believe to be typical of the inside of those pellets.

Q. The same thing is true of the other pictures appearing on this sheet?

A. That is right.

Redirect examination • • • [389-390]

• • • Q. One question, there may be some misapprehension about your answer. You do not know of any records, do you, that state in record form what process was used in making this sample in Exhibit 24?

A. No.

Q. Are the records as to the procedure used in making the beads commercially in Lousiana in 1931 and 1932 available?

A. That is what I mean.

Q. You simply received this from Louisiana as a sample of the product that was then made?

A. That is right.

Q. And you cannot go beyond that in identifying the procedure whereby these beads were made?

A. No, sir.

Q. As far as you know, is there anybody else in the world that can?

A. No, sir • • • [390-391].

[fol. 206] • • • Testimony of WILLIAM B. WIEGAND (Recalled).

Direct examination • • • [391].

• • • Q. You are William B. Wiegand, the patentee who testified formerly in this case?

A. Yes, sir.

Q. You heard what Mr. Drogin had to say as to the requirements of the rubber manufacturing companies as to the purity of the pelleted carbon black product they are willing to buy?

A. Yes, sir.

Q. Do you know it to be a fact that such rigid requirements as to purity as he outlined are applied by the rubber companies?

A. Most assuredly.

Q. Are they very particular about that?

A. Yes, indeed. They reject or accept on the basis of it.

Q. By referring to your patent in suit it is stated there that the product is substantially pure carbon, and I think you will find in the specifications some rather definite indi-

cation of what is meant there by "purity." I wish you would read that and state just what the requirement is.

A. Certainly "pure" in our interpretation of the word meant that it would not have been contaminated with foreign added material. I do not find the word precisely anywhere in the specifications but I do think this particular quotation most nearly explains what I had in mind in connection with that word. On page 3, line 52, it says, "In general it is preferable that the creaming agent be completely volatile so as to leave the carbon pure." We did not want any of these binders in the carbon.

By the Court:

Q. What about line 100 on page 2?

A. Yes, sir. Shall I explain what that means?

The Court: If you can.

A. Yes, sir. There are cases where grit can be normally ground up and it is unnecessary to do anything about the carbon black grit. We keep it down to a satisfactory level. [fol. 207] In the two-liquid process when we were running —I am thinking particularly or probably of some early stages when it was a batch process—we used 50 gallon drums tumbling end over end. It had this particular provision on page 2 which would not ordinarily be employed at all because the tumbling end over end would cause all the grit particles to be mixed up with it. What I had in mind was this,—that if in any particular case of especially gritty black it was presented for shipment we could allow the whole machinery to stop long enough for the grit to settle out. It was not a regular procedure; it was a provision but I do not think that was used except very rarely.

By Mr. Edmonds:

Q. Of course you were quite familiar and had been familiar for a long time with the proposals for aggregating particles with a binder?

A. Yes, I heard a lot about that.

Q. Was this reference to purity there in reference to that use of binders?

A. As a contrast to that, yes, sir; that is it exactly.

Q. On this matter of porosity you heard Mr. Drogin comment. Can you inform us further on that, if possible sum-

marizing the situation with respect to porosity, first, just as a matter of comment, secondly as a matter of demonstration?

A. Well, it is a little broad, I do not know exactly what you mean.

Q. What is the fact about the present-day commercial production like plaintiff's pelleted black and defendant's Sayre black and the pellets of carbon black made by the two-liquid process either now or back in 1930 and 1931 with respect to porosity? In discussing that refer to densities of 25 pounds per cubic foot because that is the measure that we want?

A. They are all porous. They have to be porous because they weigh much less than they would weigh if they were solid. In the second place, I think they would all show up on porosity tests, that is, they would all drink. They would imbibe. I am perfectly sure that all of them would express [fol. 208] air if it were pushed outside. If you take a sponge and you squeeze it and then you let it come in contact with liquid it will imbibe water. That is what I mean. If you take these spongy things, dry them and squeeze out with water you get bubbles out of it. That is what I meant by a bubble or expression test. I think that is true of all of them. I think that is expressed as easily as any other way by apparent density as we know it. It is the weight per cubic foot.

Q. As to any difference of structure between present day micronex and Sayre beads on the one hand and the two-liquid beads of 1940 and the two-liquid beads of 1931 on the other hand, if there is any such difference, will you state generally what that difference could be, and if there is a difference, whether it has any significance?

A. My information and my experience in testing these things is that they all show porosity; that the beads you have described are all in the range of 25 pounds per cubic foot. We have talked rather loosely about 25 pounds per cubic foot where there might be easily a 5% difference in that figure, depending on how tight the bag was. They are all about 25 pounds and they range between 25 and possibly as high as 28 pounds.

Q. Maybe I did not make my question clear. Is there any difference among them with respect to the distribution of the voids, assuming that all of them are of the same

aggregate porosity by reason of being of the same weight; is that difference of any magnitude and, whatever it is, is it of any significance?

A. I am not prepared to say. There might be some differences in the structure or in little blow holes that may appear in these pellets. I have seen blow holes in all of them. I have seen plenty of blow holes or large cavities in the two-liquid beads and I have seen them in the other beads.

Q. Sayre beads and plaintiff's beads?

A. I have seen plenty of big voids and little voids in about [fol. 209] all I have looked at. I do not attach any particular importance to those differences.

Q. You have been contacting people in this carbon black business and the use of carbon black for many years. Did you ever meet anybody else who attached any significance to that?

A. I have never met anybody in the big tire companies that even wanted to discuss it. We have talked about it ourselves but we have never had the matter raised.

Q. If the product breaks down to the final particle, as to the size or distribution of the voids that make up the porosity, would there be any difference?

A. If one product had such an extreme development, or what I call excessive porosity, that it went all through the majority of the pellets, I conceive that would show up in the customer's factory after transportation for 1000 miles, when they might be dusty. I can conceive of the customer's using facilities for measuring how much is left in a hopper car and he arrives at figures which give him a pretty good idea of the cleanliness of the product.

Q. As a matter of fact it is rather simple to demonstrate the porosity of beads of the sort we are talking about, is it not?

A. Yes, indeed, sir.

Q. You can actually make the exit of the air from the pellets visible?

A. Yes, sir, it is a very simple affair.

Q. You can do that right in the court room with a microscope, can you not?

A. Yes, sir.

Q. Also it is possible to demonstrate the taking up of liquid by porous beads, is it not?

A. Yes, the imbibition test.

Q. Are you prepared to do that in the court room?
A. Yes, sir.

Mr. Edmonds: If your Honor is interested in seeing either or both these demonstrations we will be very glad to make them. On the state of the record it seems to me [fol. 210] to be unnecessary because it seems as though all concerned are agreed that plaintiff's beads and defendant's beads and the two-liquid beads and all other beads are porous:

The Court: It is very doubtful whether seeing an experiment would be helpful to me. If he has any testimony as to the results of experiments, that would be a more satisfactory form.

Q. Will you outline briefly just what the procedure is in making the exit of air from these pellets visible, and in that connection state whether you have done it and have seen the air bubbles and have shown them to others and caused them to see them, even one whose experience in the use of a microscope is as little as mine?

A. Yes, we have done it often. The "expression" or "bubble test" is done by the microscope by having a slide with a little depression in it, putting in a few drops of linseed oil, for instance. You will see they sink, they get wet, and you will see, in a sort of lazy fashion, a whole stream of bubbles coming out of each pellet.

By the Court:

Q. That demonstrates that but it does not measure the degree?

A. No, excepting so far as you can see a great stream coming from all of them. It is not an exact and definite mathematical test, it just shows they are full of air.

By Mr. Edmonds:

Q. You have done that with United beads and plaintiff's micronex beads, two-liquid beads and various others?

A. Yes.

Q. You have done it in the past few days and shown it to others?

A. Yes, indeed.

Q. Of course the bubbles are magnified and look rather large?

A. They are large.

Q. They look like glass beads?

A. Yes.

Q. Nothing in that test indicates any difference whatever between the United beads, plaintiff's beads, two-liquid beads and others?

[fol. 211] A. They all provide a very beautiful stream of bubbles. That is about all you can say.

Q. Will you tell us about this other test that illustrates the air coming out?

A. The linseed oil?

Q. Will you tell us about your test which is based on the entrance of the liquid into the voids rather than the exit of the air?

A. I would like to have one test tube while I am explaining it?

The Court: That has been explained. You put the pellets into the test tube to see how it is broken up.

Q. Have you performed this other operation within the past day or two?

A. Yes, sir.

Q. In performing it you used bird shot about the size of pellets?

A. Yes, sir.

Q. There were four tubes; do you remember what pellets were in those four tubes?

A. In one there was bird shot of about the same size as a pellet. In No. 2——

Q. (Interposing) Micronex beads?

A. Yes. In No. 3, Exhibit 20, Sayre beads, Tube 4 contained Exhibit No. 11, two-liquid beads. Tube 5 the two-liquid beads made by Dr. Drogin. We took the test tubes and put into them 2 cc. of acetone. Then we simultaneously inserted all five cylinders until they touched bottom. These five cylinders have a little bit of cheesecloth on the bottom to keep the black from blowing out and a little acetone. There was a little wetting action, that is, there was a little capillary rise in each tube, even including the bird shot, corresponding to the weight of the actual pellets. That occurred very promptly. When that was complete the bird shot test tube showed about only 1 cc. of acetone still left in. During the next minute or two all of the tubes showed the liquid filling as it was sucked up through the cheesecloth screen at the bottom, and up into the inner

cylinder containing carbon black. All of the four samples imbibed the acetone at about the same rate. At the end of three minutes that was done. Incidentally that time varies a little bit. If it is a warm day it takes a little [fol. 212] different time than if it is a cold day, but in about three minutes all of the black had sucked up all of the liquid and there was none left in those four tubes. The height of the acetone in tube No. 1 did not vary so of course there had been no change.

Q. It did not change for a long time thereafter, did it?

A. It did not change at all.

Q. That level of the liquid in the test tube containing bird shot is the level assumed as the result of the balance between carrying the water up and the pressure down?

A. Yes.

Q. The fact that the water or acetone level remained stationary after a very short time in that tube and went down and out in all the others——

A. (Interposing) It was sucked right into the pellets in the other cases.

Mr. Edmonds: That is a demonstration which we can give in the court room at any time if anyone wishes it.

Q. You heard Mr. Drogin tell about his procedure in making beads by what he said was the procedure recommended in the specifications of the patent in suit. I wish you would state your views as to the procedure that he used and whether or not it was fair practice of what the patent teaches.

A. Drogin's pellets were not pushed around enough. When you make pellets by this two-liquid method you have to beat them up. That is why we tried in the patent to convey that general information. There is a great deal of difference between stirring things gently around or even quite violently and turning them over end over end as we do when moving like that (indicating). You get a different result. If you use adequate tumbling and sufficient blows or impact you get a pellet which is pounded down to what it ought to be. That is particularly defined as a pellet which is dense enough to weigh about twice what the ordinary carbon black weighs, that is to say, about 25 pounds per cubic foot. Any deficiency in beating up these pellets show low apparent density and low weight per cubic foot. [fol. 213] We have made them by shaking, we have made

them by tumbling over. On page 3 of the patent we show what we use and how it is worked.

I think perhaps it might not be amiss for me to explain why this five minute definition appears on page 2. It is true the patent mentions five minutes but when we make pellets in five minutes they get a terrible shaking in a very small container only about this big (indicating). You can make them that way if you shake hard enough.

By the Court:

Q. Where does that appear?

A. On line 75: "The mass may be thoroughly shaken or otherwise agitated for about five minutes." If that is casually done or inadequately done you do not get the condition of density otherwise mentioned. It is to that I attribute the low density of Dr. Drogin's pellets.

By Mr. Edmonds:

Q. What have you to say about the product that he got? For your use in that connection I will show you a sample of his product, Defendant's Exhibit 13,—a little tray.

A. May I ask if these are as made or after screening? Were they supposed to have been screened?

Q. Where is the rest of Exhibit 13, is it on the table here?

A. Those are poor pellets. They are not well shaped and they have a very different kind of shape to them. I would not call those very good beads.

By the Court:

Q. You do not think they are very good looking?

A. No, sir, I do not.

By Mr. Edmonds:

Q. Is that as much as you can state about the beads from looking at them casually?

A. They look like rather poor beads. I would not care to say any more about them pro or con.

Q. Something was said about the process described in the patent; some suggestion that it ought to be used as a batch process or would have to be. What is the fact about that?

A. We started with the batch process but we discontinued and used a continuous drum method. That is the agitation

[fol. 214] operation that was actually used when the commercial production began in the field,—that was continuous.

Q. The patent itself states that the process may be practiced as a continuous one,—down at the bottom of page 3.

A. "For continuous operation the agitation may be by any means which will impart rapid changes in the direction, etc."

Q. Also this book entitled "Flow Carbon," which the defendant puts in evidence as its Exhibit No. 20, states it is continuous.

A. Along broad lines, yes.

Q. I hand you a little booklet; will you identify that if you can? If you have any pride of authorship you might say who wrote it. Also please tell when it was printed and distributed.

A. This is a technical bulletin published in 1931 by Binney and Smith with whom I was then connected. I think I was the author, in fact I know I was.

Q. It has a copyright notice on it, does it not?

A. Yes, sir, 1931.

Q. Were copies of that printed in any considerable number and distributed among the users of carbon black?

A. I would say at least 1000 or perhaps more.

Mr. Edmonds: I offer the pamphlet in evidence as "Plaintiff's Exhibit No. 26."

(Plaintiff's Exhibit No. 26, filed.)

Q. Here is this diagram, Plaintiff's Exhibit No. 6, which has some relation to the procedure the defendant uses at Sayre in making its pellets. You understand, I take it, the respects in which this, being diagrammatic, is a departure from the actual, do you?

A. I do, yes.

Q. The cylinders actually are longer. This second one is below the first one and the travel is from left to the right in the upper one and from right to left in the lower one?

A. Yes.

[fol. 215] Q. That has all been cleared. As for the actual construction employed in those cylinders it is as illustrated in Defendant's Exhibit No. 8. You recall that, do you not?

A. Oh, yes, I remember that.

Q. Is there some distinct feature of an operation of that sort that results in the production of pellets in the operation

of such apparatus as that? If so, will you tell what it is and when, how and under what circumstances knowledge of it came to you?

A. The single liquid process—may I inquire if that is it? The real way it operates—I can give you my idea of it. When the black is wet down with water you get a different thing, depending on how much water you use, and at a certain stage of adding the water to the particle it passes through a very interesting number of physical stages. It is hard to describe the condition. With carbon black containing about its own weight of water or a little more, it packs like a snowball in your hand. It has almost the possibility of packing it. That is the raw material of the single-liquid process.

My understanding of that whole single-liquid process is that, depending on the exact physical atmosphere of the black and water and depending on the amount of water added, that to get pellets you can adjust the whole scheme of agitation so that it is exactly suited to this particular type of mixture you are dealing with. You have to have commotion in the cylinder and that commotion must be of a kind that will generate and build up and eventually perfect these little pellets. To boil down this single liquid process to its essence, I think you require some kind of agitation that is perfectly adapted to the motion of commotion or agitation so that it is in ideal balance of texture,—the black and water that you are using * * * [391-407].

* * * Q. I believe you had not finished your answer when we adjourned, Mr. Wiegand?

A. I was trying to give an idea of the principles behind the single liquid process for making beads, and I think I said that it involves a certain adjustment, a nice point of [fol. 216] adjustment between the operating conditions, the amount, time, nature of agitation and the length of time of the exposure of the mass, the dough mass, to this, and I think I could do no better really than just say that it lies in what one might call a perfect corrolation between the conditions and the mixture of carbon black and water that determines the success of a single liquid process of that kind.

Q. State where you first saw that operation in use and what gave rise to its use there.

A. The single liquid process, when I first knew it, was in 1932, I think, in our company. There was a man, the

late Mr. Herman Glaxner, who was raised in the carbon industry, and who was, in my judgment, one of the most brilliant mechanical geniuses I have ever known. He was in charge of our two-liquid plant down there, and he had to load in this aviation gasoline and go through all this continuous process of making beads, and I think one of the actual reasons he took such an interest in the process was that he was dizzy or, as he used to say, he was drunk all the time with this smell of aviation gasoline.

Water had been dammed as a material for making beads for many years, and I think, perhaps, Glaxner's great contribution was that he was not discouraged by that. He had the pellets there and he wanted to get rid of this two-liquid method for various reasons. He devised a scheme whereby he could, by properly selecting the range of water that he added, combined with this mechanical arrangement adjusted and perfected to suit that, make beads. Actually, in retrospect, I think the secret was that he kept the water ratio below a point where the free liquid level was reached. He did not use a thin paste, but used what I believe he called a dough, and that in combination with the proper type and duration of agitation gave him pellets which were identical in their properties wth the two-liquid ones.

I have always considered that contribution of Glaxner's as a major one, because it looks so simple in retrospect, [fol. 217] and yet it was something none of us ever thought of at the time we were concerned with the problem.

Q. He did put it into use there in the plant, did he not?

A. Oh, yes, in 1932 he put it into use.

Q. And you have been using it ever since?

A. Using it with almost no changes.

Q. Is that quite adequately illustrated and described in this Glaxner patent, re-issue No. 21,379, a copy of which has been offered in evidence, and which is included in Exhibit No. 1 for the plaintiff?

A. Yes, that is the Glaxner single-liquid process all right.

Q. Now, Mr. Wiegand, there are a number of patents that have been put in here by the defendant, and I am going to ask you to refer to quite a number of them; but I think as to some we can deal with them in groups.

There are two patents in evidence, Defendant's Exhibits Nos. 22 and 23, that relate to pills, that is, medicinal pills, the Carter patent No. 207,013, and the Metcalf patent No. 671,804. I think it is hardly necessary to refer to them.

The same is true of two patents relating to matches, Grossman No. 1,422,698, and Ikeda No. 1,496,314, and that brings us to a rather considerable group of patents which you have told us relate to the use of binders in various substances, among others carbon black.

Perhaps you can give us some general statement that would be helpful, dealing with binders generally, in pelletizing; that is, a general statement that will have application to all of these patents in this large group which you say disclose the idea of using a binder for use in agglomerating, some of these patents relating to carbon black and others to such things as coal dust and explosive powder and metal ores, cement and so forth.

A. Well, the rubber industry began about, I suppose, 1914 to use large quantities of carbon black, and for four or five years I don't think we had any clear conception [fol. 218] as to why it worked as it does. They knew it gave better road mileages, but the mechanism of it I think was not clearly understood, but it became gradually more and more evident that the whole secret of carbon black reinforcement lay in its enormous surface development, which in turn was due to the fineness of the particles.

Now, therein lay, I think, the reason why binders did not work. If you took a solid binder, something of a plastic nature that can be put into solution or added in a molten condition and solidified, you found that the same thing carbon black did to rubber, namely, to strengthen it by gluing the particles of rubber together, worked also with the binder. In other words, the carbon black attached itself to the binder in much the same way as it does to rubber.

It has been shown, as a matter of fact, that carbon black reinforces asphaltic compositions in a manner quite analogous to the way it behaves with rubber. The reason for that was that the use of any solid binder simply glued the particles of carbon black so closely together they would not disperse any more in the rubber.

Now, getting away from solid binders to liquid binders, I think the category of tars is probably the most favored. There you were up against another aspect of the fineness of subdivision of carbon black. If you wanted enough binder of a liquid character in carbon black to be effective, you had to add an enormous quantity of it.

A pound of carbon black covers twelve acres, so if you want to bind a pound of carbon black together with tar,

you have to, in effect, spread the tar over twelve acres. That takes a bit of doing and takes quite a bit of time. We have used up to two hundred per cent. by weight of tar on the carbon black in order to get it into a non-flying condition. Randall came along, for example, and cut that down quite a bit. Randall said, "If I have the tar in naptha, I can spread a much smaller quantity of tar around the same amount of carbon black," and he did, and he cut it down from two hundred per cent. to twenty-five per cent. by [fol. 219] weight on the black. There, however, a whole group of new problems arose in rubber.

If you build a tire tread with twenty-five per cent of tar on the carbon black, you finish with twelve per cent of tar on the rubber; altogether too much. The amount of softener used in an automobile tire, the tread part of the tire, is never in excess of about two or two and a half per cent, so that the very minimum amount of liquid binder that can be put into carbon black and still be effective is still about five or six times too much to be tolerated in the final rubber compound. It would weaken it.

Now, I think those two points of view cover essentially the reasons why binders, whether solid or liquid, were never wanted and never tolerated by the rubber trade. I could mention others—take a white shoe, for example, the sole of which is black, and which contains a piece of white rubber around the edge called foxing. If you had tar in the black part and then put white rubber on top, then you got the tar bleeding through, as they call it, diffusing through and staining the whole of the white rubber.

Q. Aside from everything else, will the rubber man allow the carbon black man to tell him what is to go into the rubber, or to have any control over his formula, as for instance, by mixing something in with the carbon black he sells him?

A. He will not, and I think the main reason is that he has so many formulae.

Q. In the thousands; isn't that right?

A. In the thousands, yes, and what might be all right for one would be hopelessly wrong for another.

Q. There has been some mention here of the Geer patent No. 1,245,700. Is that properly classified as a binder patent?

A. Well, I think it comes under the category of that second group I mentioned, because it had an oily material, kerosene, I believe it was, added to it, and that was carried right through into the rubber, so it must have been a binder. [fol. 220] Q. It had to go into the rubber if it was to have the effect for which he put it in, namely, to allay dusting, did it not?

A. Well, that is true, because Geer used fifty per cent of liquid on the black, and that amounted to—that amount of liquid on the black, unless left in, just would not do anything to the dust problem.

Q. And he distinctly states that the binder is in the carbon black, not only when the black goes on the rolls, but also when it goes into the rubber, and into the finished product; does he not?

A. In part or in whole. He provides—he tries, rather, to secure a partial evaporation during the mixing operation, but admits that the rest of it goes right through the finished rubber.

Q. And what would be the effect on the finished product of any such procedure as that?

A. Well, it depends a great deal on the kind of rubber goods being made. I don't think Geer's process was ever adopted, so I have no way of knowing what kind of rubber goods were actually produced, because I don't know of any that were produced by the Geer method. What I have done was to duplicate the Geer process and I have here a sample of the kind of rubber one gets from it.

Q. Will you state just what that is?

A. This is a rubber compound containing thirty-five per cent by weight of carbon black to one hundred parts of rubber, and these three experiments represent, in the first place, the control sample which was pure carbon black, commercial carbon black, and in the second case the same thing added in the same amount, but containing fifty per cent by weight of kerosene mixed together and milled in, and in the third case, fifty per cent of water and the resulting rubber was vulcanized in open steam, I believe forty pounds of pressure, something like thirty minutes—a normal time for this particular formula—and it blew badly, by which I mean that huge porosities or bubbles, if you want to call them that, occurred in both cases where the foreign material was put into the black.

[fol. 221] Q. Is that visible on these samples you have there?

A. Yes, I think it is quite clearly visible. Actually the water blew worse than the kerosene.

Q. Then, these two samples show the large pores which appear to the naked eye in each of the two experiments?

A. That is right.

Q. No such procedure as that is used in making rubber for tires, for instance, or anything else except where you want that condition of porosity, such as you might want in a sponge; is that right?

A. That is true. Those conditions of porosity can be in part mitigated, if special provisions are taken. If you know ahead of time that you are going to get porosity of that kind, you can take precautions which would mitigate it. I don't want that to be on the record as my idea of it, as to what would always inevitably occur, but that is typical of what is done.

Q. But that represents what you say would occur in practicing the procedure of this Geer patent?

A. That is exactly what happened when we practiced it, yes.

Mr. Edmonds: I offer in evidence this card with the three experiments on it, as Plaintiff's Exhibit No. 27.

(Plaintiff's Exhibit No. 27, filed.)

Q. Mr. Wiegand, there has been some mention here of the Knowlton patent No. 1,286,024. At the moment, I will just ask you if in that patent the use of a binder is recommended.

A. Knowlton and Hoffman gave, in the only example I can see in the patent, the following instructions: "Take 220 parts lamp black, a thousand parts of hot water and five of starch, the starch being first dissolved in the water." In other words, they choose starch as a binder.

Q. That starch would be a binder and is there for that purpose?

A. Yes, sir.

Q. There has also been some mention of the Lewis patent No. 1,236,082. Does he recommend the use of a [fol. 222] binder?

A. Yes, he says that in certain rubber formulae, certain waxes and oils are used. "In my improved process I make

an emulsion of these ingredients with a liquid, if they are insoluble in that liquid, or dissolve them in the liquid if they are soluble. A paste may be made using four to five times the weight of the liquid to the weight of powder, and as the hydrocarbons or other substances are thoroughly intermixed with the liquid, they will thus become thoroughly intermixed with the powder." That, I think, is a pretty fair description of the use of a binder.

Q. He is referring to binders above there in the paragraph on page 2, lines 18 to 27, too, is he not?

A. Yes, that is another reference.

Q. Do you remember the Sheppard patent, No. 1,651,733? Is that a typical binder patent dealing with carbon black?

A. Yes.

Q. I think you can speak of that very briefly. You might state whether it is closely related to the Randall Patent, No. 1,405,439.

A. Yes, Sheppard saturates the carbon pigment with a vulcanization accelerator.

Q. Some of these patents, these binder patents——

A. I beg your pardon, I think I made a mistake on that patent.

Q. Will you correct it then?

A. I had another patent in mind. I said that Sheppard saturates the carbon pigment with a vulcanization accelerator and that is wrong. He coats the carbon black with wax.

Q. The proposal there in that Sheppard patent is much the same as Randall's proposal, is it not, to use a solvent along with the substance which is the binder?

A. A solvent or an emulsion. He uses it to spread the binder out a little more thinly, yes.

Q. Now, taking up next a couple of other patents in this binder group, in this case patents dealing with substances other than carbon black, I wish you would refer to those two British patents of Trent, Nos. 151,236 and [fol. 223] 183,430. I think you can deal with them together because the two are so much the same, the second being for carrying forward the process of the first.

A. What I have before me is 183,430. Is that the second patent?

Q. Yes, that is the second. The first is 151,236.

A. Oh, Trent was not dealing with carbon black at all. Trent was making a special type of coal briquette by means

of oil which served to keep the particles of coal together like any other briquetting operation, and I was interested in reading Trent to note that apparently that association of his coal dust with the oil is only a temporary sort of thing. This is what he had in mind, that Trent's oil-bound powdered coal briquettes could, if desired, be restored to their original powdered condition by merely getting rid of the binder. I am quoting page 4 in his Patent No. 151,236, lines 22, 23, 24 and 25: "Treating agent may also be removed and the carbonaceous particles in a substantially dry state used as a powdered fuel." That, I think, quite definitely points to the fact that Trent was preparing as a rather purer form of briquette a product consisting of coal ground in water to get rid of ash, presumably, and other impurities, and then combined with oil and at will the oil removed and the original powdered coal restored, and it could be used either in the briquette form or, as he says in that quotation, in its original powdered form.

Q. By way of summary, is all of the description in these two patents a description of an aggregate of coal dust and oil?

A. Yes, that is what it is.

Q. Have you a list of these patents in this binder group before you?

A. No, I have no special list here.

Q. Just for the purpose of abbreviating this matter of binders, I will ask you whether in addition to these patents you have mentioned in this connection, that is, the patents of Geer, Knowlton, Lewis and Randall and Sheppard and Trent, the following also come within this class of binder patents, namely, Lawrence 377,901, Acheson 1,431,079, Curbishley British Patent No. 127,770, Saunders 598,464, [fol. 224] Rodman 1,076,235, DuPont 1,106,087, DuPont 1,239,368, Payne 1,114,029, Ellis 1,116,044, Rodman 1,239,221, Thorssell 1,466,571, Schaffer 1,570,086, Wisner 1,756,896 and a British Patent of Societe something or other, No. 244,461. Are all of those patents, in your opinion, to be classified in this group of binder patents?

A. Yes, sir. I have read them all and I so classify them.

Q. Now, just by way of eliminating one or two others, what is the McMahan Patent No. 1,541,233 and what does it relate to?

A. I was in Mr. McMahon's factory when he was working on improved methods of compression. I never studied the machine in detail but I know that in McMahan's it has to do with means of compressing carbon, a machine for doing it, rather.

Q. Nothing else to it but just compressing it, is there?

A. No. I knew about it at the time.

Q. As a matter of fact, the machine did not work very well, did it?

A. I don't think the machine was ever adopted.

Q. Now, look at a couple of patents that follow in another group, the Davis Patent No. 1,191,673, first. What part of the operation of making carbon black does that patent deal with?

A. That Davis patent is essentially an agitating machine, deals with an agitating machine, that is, Davis densified the black by stirring it. However, he does emphasize the fact that one of his major objectives was to destroy these flakes which characterize black as it comes from the channels, so his machine also had the objective of breaking these flakes down so that the whole black would be fine enough to go through bolting sieves.

Q. It is quite plain that beyond his agitator the black comes to a very fine screen through which it has to pass, is it not?

A. That is right.

Q. And one of his purposes was to stir it so as to get out some of the air?

A. That is right; to densify it and get it in shape for passage through the purifying screen.

[fol. 225] Q. Another patent is the Bartlet Patent No. 809,354, which is more or less related to the Davis patent. Will you make a brief statement about that?

A. Well, Bartlet apparently did the same thing to two other things that Davis and others do to carbon black. It is a process of agitating metallic salts and oxides quite apart from any references to carbon black. His primary objective is to eliminate the greater portion of the air and gas contained in such pulverulent masses.

Q. And densify the mass?

A. Yes.

Q. Like Davis, except——

A. (Interposing) Well, it is a little different mechanically, I would say.

Q. But the idea is the same as Davis', except Davis' machine was described as applied to carbon black?

A. That is right.

Q. We come now to some remaining patents about which a little further detail should be given. Will you take first the Evans Patent No. 1,175,732?

A. Well, Evans treated coal pitch in order to get out of it a somewhat purer form of carbon. To do that he treated it with hot alkali and followed by extraction of impurities by means of organic solvents. The end product was a purified carbon dried out into a caked condition, page 2, line 55 defining his end product. I do not see any relation between this and carbon black.

Q. He seems to characterize his final product rather definitely on page 2 in lines 98 to 101. You might read that and state what his summary is.

A. (Reading) "It will thus be seen that this process yields at will either an absolutely pure carbon in a finely divided state or a carbon containing a small quantity of bitumen which renders it suitable for certain special purposes," is what that says.

Q. That bitumen in there, what function does that serve, Mr. Wiegand?

A. Well, it would have to be a binder.

[fol. 226] Q. So the product is either in a finely divided state or in a state in which the fine particles are held together by a binder?

A. That is right.

Q. Will you state whether or not you are familiar with the Pike Patent No. 1,285,363, and will you turn to that patent, please?

A. Yes, the Pike Patent No. 1,285,363 describes the purification of the by-product carbon from oil and gas manufacture. This is a very low grade product with which I happen to be familiar, normally sold as fuel. He washes out the tar and then roasts out the volatile hydrocarbons and the resulting solid, cake-like mass is then ground to induce the powdered condition. The only reference I can see in this Pike patent to a pure carbon is one in which it is in a finely divided condition. The only pellets which I have seen mentioned in Pike were composed of tar. For example, page 3, line 32, he does mention pellets there. He says, "The remainder being in pellets and flakes about

the size of a pea or ten-cent piece," which he says are "composed largely of tar."

Q. Please look at the Weinreich Patent No. 1,184,397 in the exhibit.

A. Yes, Weinreich is concerned with restoring filtering media by heat activation. The only black he mentions is bone black, which is the charred remains of animal bones, the carbon content of which is a very minor proportion. Now, Weinrich provides for heat activation of what I take to be spent bone black, or spent media, to restore its activity.

Q. Spent bone black is bone black after it has taken up impurities and performed the function for which it is used, is it not?

A. Yes.

Q. So you want to take those impurities out of the bone black so that it will be available for future use?

A. Yes. He does that by means of heat, which is a well-known way of achieving that particular end.

Q. What about the product; is it at all times particles of bone, both before and after use and after reactivation, [fol. 227] and at all other times?

A. Well, except for its improved activity, I would say yes.

Q. He seems to subject those particles to rather high temperature. Does that suggest the possibility of getting anything out of that patent that would be applicable to carbon black?

A. Well, if the temperature was anywhere over 500 degrees I should say it would be wholly inapplicable. I don't see the temperature reference.

Q. Look at the second column, page two.

A. I have that.

Q. That indicates the temperatures.

A. Yes. We try to avoid conditions like that in carbon black industry; danger of burning it up.

Q. He puts his bone black through a big rotating cylinder which is heated from an exterior furnace, does he not, according to this patent?

A. That is right, to facilitate the uniform distribution of the heat treatment, heat activation.

Q. Next please look at Spearman Patent No. 1,377,397 in the exhibit.

A. Well, Spearman deals with a flotation process for separating graphite from its—

Q. I did not understand you.

A. I say, Spearman deals with a flotation process for separating graphite. The ore is ground in a ball mill with water, and then an oil such as kerosene added. The particles rise to the surface, covered with a film of oil. He provides that this oil can then be driven off by heat, whereupon the graphite particles and flakes, he says, "resume their discrete association." I assume he means discrete condition. I do not quite understand why a man would say "discrete association," though. Page 2, line 52, that is. Also page 1, line 96, he says, "On being dried"—in other words, the binder removed, the oil film—"powdered graphite falls away from the flakes and can be separated by screening." Spearman does not talk about carbon black. I do not see any references to pellets. I do not see any [fol. 228] references to a dust problem anywhere in this particular patent. I have noted under Spearman that it appears to be his primary objective to recover graphite flakes in their naturally occurring state.

Q. Next please refer to the Brownlee Patent 1,478,730. That, I think you will find, does refer to carbon black, does it not?

A. Yes, indeed, that is a very significant patent in our industry and marks, I think, the first important departure from the so-called impingement process for making carbon black type of pigments. In other words, Brownlee does not use flames but he makes his black by heating up a checker-work to 2500 degrees and putting natural gas through that, thus breaking it down into carbon and hydrogen. It is a very efficient process chemically speaking. It gets probably at least five times as much carbon out of the gas as we do when we impinge flames, but as a result of that and following, I am sorry to say, the inflexible rule of our industry, the more you get out the lower the quality, and this black (which is, in modified form, now on the market known as Thermax) differs from true carbon black, or as we sometimes call it, channel black, in that the particles are very much larger. In the second place, the carbon is, practically speaking, nothing but carbon. There are not any gases combined or associated with the carbon and the result of those two features is that this Thermax is much denser, and I am referring to apparent density there. You can pack more than twice as much in a given size package. The black is gray, oily in texture and that, I think, explains

the physical differences mentioned by Brownlee in his patent.

Now, the Brownlee process has been in continuous use since this patent was taken out, or shortly thereafter, and improvements have been made in the Brownlee process by feeding in natural gas through their furnaces. In a diluted form, with four parts hydrogen to one part natural gas, they have recently produced a very much finer product, not as fine as channel black, however. Now, this is not [fol. 229] quite as heavy as the original Brownlee product. It is more finely divided. It does not present any great dust problem, I must say, but within recent years the great trend toward pellet black and bulk handling have induced the manufacturers of this patent, you might say a Brownlee offspring, to go into pellets too; and the owners of this whole patent picture have very recently completed a license agreement with our company in that connection.

Q. For a license to them under this Wiegand and Venuto patent in suit?

A. Yes, that is right.

Q. And they expect to be in production of pellets very shortly?

A. I have no information as to that. I know they are working on the thing and developing it, but I am not able to state just when they expect to go into full production of it, however.

Q. Will you next please refer to the Spear Patent No. 1,511,984. This is another patent dealing with carbon black, is it not?

A. Yes. Well, Spear decided that it was not enough to add carbon black by itself; it is not enough to add a vulcanization accelerator by itself. He is a very eminent chemist and he provides in this patent for saturating the carbon product—I presume he had carbon black in mind—with a rubber vulcanization accelerator and incorporating both at one time in the rubber. I imagine he expected a greater efficiency of behavior on the part of the accelerator because it was disseminated throughout this enormous surface of carbon. I don't know whether his patent ever got into use or not.

Q. What is the accelerator which he mentions, and I think the only one he mentions?

A. Ammonium sulphide. I think that is pretty much obsolete by this time as an accelerator. He saturates the

carbon with ammonium sulphide and then adds that to the rubber.

Q. What is the function of an accelerator in curing a rubber batch?

A. It promotes vulcanization or combination of rubber with sulphur and is an essential ingredient of practically [fol. 230] all modern forms of rubber being manufactured.

Q. Is there anything in this patent that has anything to do with the Wiegand and Venuto patent, any relevancy to it?

A. I don't see any even remote relevancy, no. It does not refer to pellets; no question of density changes. I am not able to find any relevancy.

Q. There is another Spear patent, No. 1,560,488. Will you refer to that?

A. This patent, I note, was filed a day later than the other one and apparently he continues the idea of using carbon black as a carrier, or other carbon pigments as a carrier, but in place of saturating them with one accelerator he takes two different lots of carbon or lamp black, charges each separately with two different vulcanizing agents and then adds them to the rubber. As he says, "I may charge one quantity of the carbon with ammonia, the second with a gas such as hydrogen sulphide, formaldehyde or carbon dioxide." He may very well have had in mind the fact that he could add two accelerators separately in this way and prevent excessive scorching, as we call it, or pre-vulcanization. Here again I am not able to discern any relevancy to the problems which we have been concerned with these days.

Q. Will you next turn to the Coffin patent, No. 1,561,971, please?

A. Yes, I have it here.

Q. Will you state what the method of that patent is essentially, and whether or not there is any application to carbon black?

A. Well, this is a drying patent and it has to do with drying clay, as far as I can make out, and the inventors point out that the standard way of drying clay involves quite a few steps. You have to make a slip or slurry, then you remove the grit, then you filter it, then you dry the clay, they say, in lumps. Then you ship those lumps to storage. Later when the material is wanted you dry them a second time, then you have to go to a high-speed pulveriz-

ing machine and get the clay back again to its powdered condition, and what Coffin and Keen did, or tried to do, at any rate, was to short circuit most of those steps. They [fol. 231] say the object of their invention is to provide a rapid and economical method of drying a pulverulent material such as clay, and what they did was to spray it down in this spraying machine and get a very fine granular form of clay, but they said it looked like fine sand, which avoided the necessity for going through all these steps.

Q. You referred to a slip or slurry. What is the meaning of that term?

A. Just an aqueous suspension of a pigment. I think Coffin and Keen stuck to clay in their work, but they did mention that they thought their process could be used with two or three other powders or pigments, of which I note they mentioned only three: barytes, lithopone and whiting. Now, these are all wet process pigments apparently. Lithopone is precipitated in water and the other two, I believe, always are levigated in water, so I think Coffin and Keen can be said to be dealing with the drying of, particularly clay and several other of these typically wet process pigments.

Q. For the purpose of drying?

A. For the purpose of drying. The word "drying" occurs sixty-six times, apparently.

Q. You might consider the possibility of making use of any such procedure in connection with carbon black; if anybody had considered such a thing, what would you say his mental operation on that subject would be?

A. Well, carbon black is dry to begin with and it does not require any powdering to get it in shape for rubber mixing.

Q. Well, had the carbon black art been through the process of considering the wetting of carbon black, and did it know by that time what the result would be?

A. Well, I suppose you are inquiring as to whether or not carbon black men would be inclined to put it into a wet condition. No, that would be ruled out because the industry had been through that. They had been through that, to wet carbon black down. Geer tried it, Knowlton and Hoffman tried it; and Coffin and Keen, as I understand it, spray this slip or slurry and that would require that the [fol. 232] carbon black, if that were used, would have to be

reduced with about at least, I would say, eight parts of water to each part of carbon black, double the ratio actually used by Lewis and Knowlton and Hoffman. Now, when even five parts of water with carbon black is wet down and allowed to dry out into a mud cake it is sufficient to injure the rubber properties, and I would say a rubber man who was asked to contemplate wetting carbon black with eight parts, or anywhere near it, and subsequently drying it out would just shudder at the thought.

Q. The process of drying they propose is one of applying heat for the purpose of evaporating the water. Have you noted what degree of heat they propose, and you might consider what such degree of heat would mean in connection with carbon black.

A. Well, Coffin and Keen, I believe, frankly state that they deal only with inorganic or non-combustible types of pigments, and it is quite obvious that is what they had in mind because on page 2, line 80, they note that their drying medium of hot air and gasses is introduced at a rather high temperature, from 500 to 700 degrees Fahrenheit, which produces a very thoroughly dried product. Now, if you did that to carbon black you would get more than a dried product. You would get a burned product. It would burn it up.

Q. Would there be any danger of something more than burning the product?

A. Well, we do know that carbon black when wet down and put in the tray, for example, and placed in a burning house at temperatures at or below 500 will burn up. Now, if you got up to 700 in the course of spray drying, if such thing were done I should think that the fine particles of carbon black floating around in this chamber exposed to 700 degrees of hot air would make one feel rather cautious about an explosion.

Q. I wanted you to speak a little further about the Lewis and the Knowlton patents, and as you have mentioned them in this connection we might speak of them further right now. You said that both of those patents refer to the use [fol. 233] of binders for holding together the particles of carbon black, but now I would like to have you speak of them and consider them without the use of binders, because the description might be interpreted as suggesting that the watered product be used either with or without a

binder. So take Knowlton, for instance, and I wish you would discuss that without reference to the binder that he recommends in the only example that he gives. What would be the effect of mixing water with the carbon black and drying, as he says; what sort of product would you get and how much of an impression would it make on the dust problem?

A. Well, if Knowlton and Hoffman had used what apparently Geer used, I am speaking now of the water primarily, mainly 50 per cent. on the carbon black, and if they tried it out they wouldn't get anything except just powder.

Q. A dusty product.

A. They would get a dusty powder.

Q. They themselves carry the water ratio much higher, do they not?

A. They naturally have to, just the same as Lewis. He had to use 500 per cent. water on the carbon to get a paste that would expel the air, and Knowlton and Hoffman, I believe, used 417 per cent., or about that amount. That is what my note is here. Well, in other words, if they just used the Geer proportion and tried that out they would get dust, and they had to go up with it. Now, if they had tried that out, they would get a mud cake, and I feel convinced Knowlton's dried mud cake was identical in general with what Lewis got. They stirred it up, the water or whatever they use, and got some lumps with it, but essentially they got a mud cake. When that mud cake is mixed into rubber, you just don't get the full original reinforcing effect of the carbon black that was universally known in those days.

I mean, when I say "those days," between 1920 and 1930, People had been through this and nobody talked about doing it any more, but I have some data of our own showing the results obtained. In this case the water ratio was five and one-half to one, the material was dried at 250 [fol. 234] degrees ranging up to 325 Fahrenheit, and the drying was stopped at 0.6 per cent. moisture. In other words, we did not dry the cake out bone dry, which would have made it considerably harder and harsher. We stopped at a reasonable moisture content that no man, no rubber man, would object to. No rubber man would mind carbon black having 0.6 per cent. moisture.

The tensile strength of that mixture in a compound containing thirty-eight per cent. of carbon black on rubber dropped from 3300 pounds when normal carbon black was used to 2950 pounds when this broken-up mud cake, prepared in this way, was used. The tensile product of the rubber, which is a quality index widely quoted in the literature, dropped from 180 units to 158 units in going from the controlled carbon black to this dried-out cake.

I think those results may be considered as typical of the school of experimenters who tried to wet down and dry out.

Q. When did you do the experimentation that led to that data?

A. Oh, this was done about a month ago, in this particular case. A repetition, it was.

Q. A repetition of what?

A. Of this mud cake experiment which was carried out in our laboratory.

Q. Going back now, to this mud cake, you said the carbon black would be unsuited for use in rubber. Is that because of a quality in the carbon black, which you have described as harsh?

A. Yes, sir. The wetting down and drying out school were on the horns of a dilemma. The cakes would either break down into a powder, had a tendency to do that on the edges and along the cracks when handled, and to the extent it did that it got dusty; and the other horn of the dilemma was the part that did not get dusty stayed harsh and I would say that that whole approach to the dust problem was always confronted with one or the other of those two drawbacks, and frequently both occurring at the same time with the same product. Some of it dusty and some of it harsh.

[fol. 235] Q. If you put in enough water so that on drying you produced the mud-cake condition, that would have to be broken up somewhere, either preliminary to going on the rolls or on the rolls themselves, would it not?

A. Yes, sir.

Q. And that process of breaking, would that produce dust?

A. It would in the act of loading it up, if you would manufacture it that way. To my knowledge, I know carbon black was never prepared in that way commercially,

but in preparing the cake and getting it into bags and unloading it, weighing it out and adding it to our rubber mill, I should think there would be a very considerable restoration, you might say, of the dust problem.

Q. And such portions as were not reduced to dust, what would be their physical form?

A. Mud cakes; just pieces of caked carbon in every conceivable shape, with sharp corners and angles and too hard to break. You could break them, but they did not give the full rubber properties of the carbon black.

Q. And because they were of every conceivable shape, any such thing as flowability would be out of the picture, would it not?

A. No, they did not flow.

Q. You knew Lewis and were familiar with his operations, were you not?

A. Yes, sir, very well.

Q. He was identified with your organization, I believe?

A. Yes, many years.

Q. Did anything of practical value ever come out of his patent?

A. No, I tried to get every scrap of information that was available, and I did finally find that 100 pounds of dried-out cakes was prepared and shipped to a large rubber company and the matter dropped there. I could not find out anything about it. That was the only amount of it ever made up.

Q. Now, the next patent I want to ask you with reference to is the Rungee, No. 1,783,983.

[fol. 236] A. Rungee took powdered coal, carbonized it at a thousand degrees Fahrenheit and of course got coke. Now, the coke was in a, to him, apparently very interesting condition. It was in the condition of hollow spheroids, according to Rungee. My impression of this patent is that each of these little hollow spheroids represented the mortal remains of a bit of coal that had passed through this terrific temperature of 1000 degrees Fahrenheit, and that is about the only relevancy I could discover to any of the subjects we are concerned with here.

Q. The final product, which you say is in the form of a hollow sphere, is a single homogeneous piece of coal; is it not?

A. The original piece, apparently the original little particle of coal which by this, you might say, high speed

carbonization had blown up and formed a little hollow spheroid, yes; just a piece of coke with a hollow center.

Q. And a single homogeneous piece?

A. So I gather.

Q. The hollow sphere has openings in it, "windows," he calls them, or openings into the interior; isn't that right?

A. Yes, high porosity.

Q. Have you finished on that?

A. Yes, sir.

Q. One other patent that needs a little comment is British patent 11,684, Pringle.

A. Pringle on the 14th of September, 1886, conceived the idea of purifying soot in a special way in England. In those days I think it was just a low grade of lamp black probably that he had available. I don't think carbon black was available in England in 1886. He pumped this in a form of water slurry through a pipe into a tank under considerable pressure. Spaced four inches away from the nozzle of that pipe he had a baffle under water, and the stream of slurry, soot slurry, was caused to impinge at high velocity against that baffle. Now, Pringle says that the carbon floats to the surface and, if I may quote, "Whilst the impurities settle in a tranquil bed on the bottom," and I prefer to leave him in that state of tranquility, your [fol. 237] Honor, if I may. I do not see any relevancy, in other words.

Q. Taking all these patents, or at least all of them that deal with carbon black in any way, and speaking of them collectively, would you say that they serve fairly well to indicate what was the state of knowledge of carbon black as of December, 1927, and the problem that existed in the plant of the rubber manufacturer at that time?

A. Is that problem as it then existed represented by this patent structure?

Q. Is it fairly presented by the several patents, or fairly represented by them, taken collectively? Do the patents point out what that problem was and do they serve to show what expedients had been proposed for meeting it?

A. Yes, I would say they do.

Q. Well, regarding the summation of all of those patents as indicating the state of knowledge on this subject, as it existed back there in 1927, what more was needed to dis-

pose of the problem and what was it that was presented for the purpose and how effective was it in disposing of the problem?

A. Well, in those years, at that time the rubber industry was gobbling up carbon black at a very rapid rate, and they were raising the proportions of black in the tires. They had gone, I should say, the better part of the way from 40 per cent to 50 per cent on the rubber by that time, and the problem was getting worse. The way it came to some of us who were at that time visiting the rubber trade was this continual pressure, "When are you going to give us white carbon black?" It began as a taunt, and it became an almost intolerable sort of a joke after a while. My feeling about carbon black at that time was that it was the most respected and the least loved pigment in the whole rubber industry, and I think that is the reason Mr. Kitchel presented this problem to us to work on. I don't know whether that answers your question or not.

Q. That presents the problem. Now, will you discuss the nature of the solution, and how effective it was and [fol. 238] why it was, as you say it was, so effective?

A. We had a pretty bad situation to start with in this problem. We had to turn our backs on binders. We knew we could not dispose of the black in wet form. It had to be pure black, in other words. The flying had to be restrained, and also ways made available to eliminate air and gases. It seemed to us that wetting down was obviously the neatest and cheapest way of doing it. Now, what had apparently ruined all efforts to make carbon black dustless when it was wet, was the fact that the water had to be evaporated out. We spent quite a bit of time on just what happens when you evaporate water, and it appeared that when you had a slurry of a pigment in a pan, when water evaporated out, a great many things happened that we did not suspect. The powder not only settles but it is drawn together by surface tension forces. The whole thing contracts, and it contracts slowly, so that there is time for the particles to nest within each other and stick to each other, and on top of all that we learned when you evaporate water down, millions of little vortices occur, and little centrifugal like things, and each of those produces a further sticking together of the particles.

It was what happened when a slurry was evaporating down that unquestionably caused the harshness of products

such as Lewis, Knowlton and Hoffman. We went up a blind alley in that direction. We conceived the idea of evaporating while stirring rapidly. We thought if we stirred while evaporating there would be no chance for these things to happen. Now, that was true, and we got a black that way that was not harsh. It mixed nicely with oils and everything, but it was powdery and dusty.

It was a very silly thing to do, in a way, because, on looking back, when you dry a thing out, no matter how much water you have and keep stirring while drying it, you pass through, at some time or other, the same proportion that Geer used, 50 per cent, no matter what you start with; so that was dusty.

[fol. 239] Another line of attack was to prevent evaporation from occurring in one direction so that the major part of the problem of harshness of wet-down black must be due to the fact that evaporation was occurring like that—the whole thing would gently settle and set into a cake—so we had the bright idea of putting the slurry or paste into a porous thimble, and then the water would evaporate in all directions at once, and you could not get any settling. That was also successful in the sense that the cake we got from that was soft and was not harsh.

We had eliminated the vortices and the settling and the contractions, but the material was very unattractive. It was essentially caked and on the slightest handling would break down again into powder, and so it was from that point of view, that we finally, after I suppose about a year, got the idea of a third way of getting rid of the water, and that was to push it out, or shake it out by means of an organic liquid like gasoline, the idea being to use as little as possible, just barely enough to push the water out, and naturally it would have to be volatile so as to leave the carbon black finally in a state of purity.

Well, that ultimately led to pellets. In other words, shaking away that water and putting in a little more of the organic liquid, which happened to be gasoline, it came finally to a point where this water would break out as we call it, leaving the carbon black on top, and that, when we shook it, gave us in one operation the elimination of the water and the pelleting, and that is about all there is to it. They dried out very quickly with little gasoline in them, and there were these carbon black pellets, which at the very first sight of them, obviously were the solution to the dust-

ing problem. They were absolutely clean and would flow from hand to hand, so quite obviously they would flow and all that; but there was a great deal of work left to do, because these little pellets were quite hard. They would resist rubbing between the fingers, and we didn't know what they would do in rubber compound, so it took us months to [fol. 240] convince ourselves, and years to convince the industry that these little quite hard resistant things could actually be restored into carbon black in its finely divided form by normal procedure in a rubber mill. That took years, and that is about the whole story.

Mr. Edmonds: It has been suggested, and I think the suggestion is a good one, that we should have in evidence a sample uniform with the others of the present-day micronex beads of the planitiff. I offer in evidence this present-day sample of micronex beads as made by the plaintiff. I presume you will accept my statement that they are beads of this type. I would also like to state that the beads in Exhibit 13 and the beads in Exhibit 15 are of the same texture.

Mr. Dike: Are the beads in this container you have just produced the same beads that were in the pitcher which was poured? I will say they are different in appearance under the microscope.

Mr. Edmonds: I will say they are offered to be about the same beads.

Mr. McLean: The beads in the bottle were taken out of the bag; the beads in the pitcher were taken from the plant of the plaintiff.

Mr. Edmonds: I suppose that means there is a little difference in the amount of transportation they had.

Mr. McLean: The difference in the handling I think is the only difference.

Mr. Dike: The beads in the pitcher are highly polished beads. They look as if they had shoe polish put on them. The other has a dull appearance. Probably the freedom from dust may be some solution to the highly polished beads.

Mr. Edmonds: You can ask the witness about that. The beads in the container I just offered I will ask be marked "Plaintiff's Exhibit No. 28."

(Plaintiff's Exhibit No. 28, filed.)

[fol. 241] Cross-examination * * * [408-451].

* * * Q. You did not intend, did you, by your testimony given today to change in any way the testimony you gave last Thursday?

A. Not intentionally, sir.

Q. Would you say that a carbon black pellet which is a friable grain would not be porous?

A. Friability would, I think, be a function of porosity.

Q. In other words, they would be porous?

A. No. I could take an electric arc carbon and break it and it would not be porous. Friability means breakability, but in respect to porous black I would expect friability to be influenced by density.

Q. As far as carbon black is concerned, your answer would be yes, is that not *that* right?

A. Not synonymous, the one being the property of the other.

Q. And a grain which is friable would also be porous?

A. The converse would probably be true. A grain that is porous would probably be friable.

Q. Would you say a grain which is friable would not be porous?

A. Yes. I can conceive of a grain of solid carbon black that would be friable but not porous.

Q. Does it not mean that it is easily mashable?

A. Yes, I suppose that it would have that general meaning.

Q. You would not suppose, would you, that a grain of pure carbon black would be friable?

A. Not as friable as a pellet, certainly not.

Q. As your friability increased your porosity would increase?

A. Yes, I suppose that the porosity increases the friability. In other words, porosity causes friability.

Q. How much do you think you would have to change your process to get a pellet which would not fall within the terms of the claims here?

A. It is too broad a question,—could you give me more help on it?

[fol. 242] Q. First take up the question of agitation. Give me some idea where you would have to stop agitation so you would not get the pellets. You say five minutes.

You are not talking about violent agitation. I want some idea of what you mean.

A. That is why that density thing was put into the patent application. It is impossible to describe agitation in general terms. You have to know what kind of thing you are agitating. I could not give you quantitative ideas on agitation.

Q. If you have an agitator which is not very severe and you run it for a longer time you would get the same results?

A. I do not know, but I can conceive that you would not. I think if you have a stirring operation you could keep that up until Doomsday and you would probably not get the same result as by proper agitation. I would not swear to that, however.

Q. The production of your beads is dependent on a peculiar type of agitation?

A. It is dependent on adequate agitation.

Q. What do you mean by that?

A. Enough to bring the product into the particular form as regards its general rounded character, its density element and those limitations of hardness and softness.

Q. Do you find any particular description of that particular agitation in your patent; if so, will you tell me where it is?

A. I think I can say we have shown quite a few successful alternative methods. I know that because we have used them. We actually got results with that on a very small scale with a violent shaking of a cocktail shaker. When we got to bigger operations we used a 50 gallon steel drum in this fashion (indicating). The drum is like that, tumbling end for end.

Q. Where does it say anything about it?

A. On page 3 at the bottom, line 128. They did a beautiful job on these pellets. I do not know whether you are interested in continuous agitation.

[fol. 243] Q. Did you use a paddle type?

A. I would rather not go on record as to any type. I was not as closely connected with it since it got into big production. I would not want to be quoted on it.

Q. You stated in your direct examination that you had to beat them up to get the pellets?

A. Yes, that is right.

Q. Is that the way you interpreted "violently shaking or rotating"?

A. Most definitely.

Q. That would be very mild, would it not?

A. It would depend on how strong your arm muscles are.

Q. What sort of an agitator did you use at Easton, Pa.?

A. At Easton, Pa., we used a fifty gallon steel drum. Here it is—a 50 gallon tank only about half full.

Q. How did you do it in Louisiana?

A. We started on a tumbling thing and went to this continuous operation. I could give you the descriptions of it in our book but I do not know what the inside of it was.

Q. Is it not true that if you put dry carbon black without any water or any other liquid with it, a pure dry carbon black in a tumbling barrel and tumble it long enough it will come out in small friable grains?

A. I have never tried it.

Q. Have you ever tried it in a rotating barrel?

A. No.

Q. Or a rotating tub?

A. No.

Q. You mean to say you do not know that they will come out as grains if you roll it long enough?

A. No, I have never done it.

Q. Have you never seen anything like that done?

A. I am not an expert on that.

Q. Have you seen the Cabot process?

A. No.

Q. Would you say I was wrong—that if you tumble that long enough it will roll into little pellets of carbon?

A. No, I would not for I have not tried it.

[fol. 244] Q. Have you ever taken a china plate with some carbon black on it and rolled it around?

A. No, sir.

Q. Have you ever seen it rolled up in bottles?

A. No, sir.

Q. Will you look at your patent, page 1, column 1, lines 10 and 11, and tell me what you mean by that?

A. "Carbon black, as manufactured, is such a very light fine powder that normally ten pounds or less, occupy a

package of one cubic foot." Well, I do not know what you had in mind but my interpretation of that would be normal agitation. You do not get more than about ten pounds into a bag, and that would be less than ten pounds if the agitation were cut down to something less than normal.

Q. And twice that would be twenty pounds, would it not?

A. This is a description of the ordinary package of carbon black. Twice ten is twenty—I give you that.

Q. If you will look at page 2, line 95, where you say "in shipping or storing, we find that approximately twice the number of pounds of these pellets can be placed in a container of a given size than is the case with the untreated carbon black."

A. Certainly not. What I interpret from that is that if we started with ten pounds and follow the instructions on page 2, column 2, we would finish with about twenty, and if we start with 12½ pounds we would finish with 25 pounds.

Q. Do not you at least start with ten?

A. No. It says "twice the untreated carbon black." Carbon black never weighs more than about ten pounds, and I do not think they are the same things.

Q. Is it not a fact that the Geer patent teaches the use of water as well as gasoline?

A. It certainly does not. You put water in as a very bad substitute. He had that kerosene, and as far as I know, he never used anything but kerosene. I have known Geer for many years.

Q. Does not that teach that you could use water?

A. You could call it that—you are better qualified.

[fol. 245] Q. Does it not also say, "The two liquids I have successfully used for this purpose are water and kerosene?"

A. Yes.

Q. Why did you leave out water?

A. Because he actually prefers kerosene and actually used it.

Q. You would have it appear that he never used water at all?

A. No, I would say if he did his job with kerosene—and he probably played quite a bit with kerosene—he put the water in there to make sure it was not left out.

Q. Look at the note in the Hoffman patent. Did not Knowlton and Hoffman also say you can use water without any starch as a concreting agent? He does say we might use water or other volatile liquid alone as a concreting agent?

A. Yes.

Q. "First mixing enough of it with the lamp black to produce a dry condition?"

A. That is right.

Q. He did not intend to have the water go into the rubber?

A. I agree with you completely. Knowlton and Hoffman were clearly desirous of avoiding things which ruin the Geer effort.

Q. In talking about Knowlton and Hoffman, will you tell me again what proportion you are talking about of water in carbon black?

A. In getting the results?

Q. Yes.

A. We use 5½ parts of water to one of black. It is a bit higher than Knowlton used but exactly the same as Lewis used. We thought that would be a pretty good cross-sectional idea of the "mud cake" principle.

Q. You were trying to make mud cakes?

A. No. I call it that because I know of no better way to describe it.

Q. Why did not you use practically four parts of water to one of carbon black?

A. Because Lewis used five. They are both mud-cake advocates.

Q. You did not follow that?

A. We departed to that particular extent, that is right.

[fol. 246] Q. When you used the pieces of rubber which form part of Exhibit 27, tell me what you did with that after you put the carbon black in it.

A. After we put the carbon black into the rubber?

Q. Yes.

A. Those samples were milled with both water and kerosene, then sheeted to cure with open steam.

Q. How long were they milled?

A. Normal milling time, in our case 12 to 15 minutes.

Q. Mr. Geer was a pretty good rubber man, was he not?

A. A very good rubber man.

Q. Do you think Mr. Geer would have not milled his rubber long enough to get results like those?

A. We took care of that.

Q. I asked you a question and I would like to have you answer that.

A. You have put me in the position of commenting on Mr. Geer being good or bad. I say he is a good rubber man. If you ask me specifically, I think he probably tried to mill it a little longer.

Q. Do you think he would have been satisfied with results like those?

A. No; that is why the patent was never worked.

Q. Do you think that Mr. Geer, as a good rubber man, would have filed a patent application knowing his results were like that?

A. I do not know if he filed it, but I think he very definitely tried to mill out a part of that volatile binder. I am sure he did. I would like to quote the results when we tried it.

Q. I suppose you will agree that both carbon black and coal are carbonaceous materials, will you not?

A. I certainly would.

Q. Do you consider kerosene a binder?

A. Yes, sir.

Q. Even when it is evaporated?

A. I tried to evaporate kerosene; it took six hours at 250 degrees F. to get three-quarters of it evaporated down. The temperature on mixing rubber is much less than that. [fol. 247] I do think the extra milling that anybody like Geer could impose on rubber would remove much less than that. I do not see how you could get the kerosene removed by milling. We mixed it and the tensile strength dropped to 2625; then we milled thirty minutes longer, allowing the kerosene to evaporate as freely as it wanted to, and the tensile strength raised from 2625 to 2750, but the original value of 3750 was in no way restored. I think there you have the history of the failure of Geer.

Q. You did not try that with water?

A. It is almost impossible to mill a batch of carbon black with 50% water on the black. It is a very impracticable mixture. We milled it and had trouble with it.

Q. Did you try milling any of the Knowlton and Hoffman pellets with water?

A. I have never seen a Knowlton and Hoffman pellet in my life, so we certainly did not do that.

Q. So you have no data on what the effect is on the Knowlton and Hoffman pellets if made with water?

A. You mean "mud cakes"?

Q. I said friable granules. I asked your if you ever tried milling such a substance into rubber?

A. No.

Q. Where do you find in column 1 or 2 of your patent anything which says that the carbon black shall not be operated wetted with water?

A. I do not find the slightest reference to that.

Q. Were the Knowlton and Hoffman friable grains or lumps, or as he describes it, dry granular lump carbon black substance?

A. Certainly not, because they used a starch.

Q. If they were made with water?

A. If they were tried out that way I would expect they had trouble. I want to say that the Knowlton and Hoffman lumps were actually described as looking like an ash in irregular lumps. That is what the Knowlton and Hoffman looked like.

[fol. 248] Q. Is soot carbon black?

A. Carbon black is soot but soot is not necessarily carbon black.

The witness was then excused.

Mr. Dike: We would like to put in specimens of beads from the pitcher offered in evidence and specimens of beads from the pitcher which was used in the pouring demonstration on Wednesday, Defendant's Exhibit No. 38. The beads are standard micronex beads brought from the plant, where they are produced, in a can. * * * [451-463].

Portions of Plaintiff's Exhibit No. 1—Patent in Suit.

Patented Nov. 29, 1932 1,889,429

UNITED STATES PATENT OFFICE

WILLIAM B. WIEGAND, OF SOUTH BEACH, CONNECTICUT, AND LOUIS J. VENUTO, OF NEW YORK, N. Y.

CARBON BLACK AND PROCESS OF MAKING SAME

No Drawing. Application filed December 2, 1927. Serial No. 237,330.

The important characteristics of carbon black which render it particularly valuable in the manufacture of rubber compositions, phonograph records, various wax and resin compositions, printers ink and other preparations, are its extremely fine state of subdivision together with the very essential property of being readily and uniformly dispersed in rubber, waxes, oils and the like. Carbon black, as manufactured, is such a very light fine powder that normally ten pounds, or less, occupy a package of one cubic foot. It is accordingly expensive to pack, ship and store such a light bulky material, and handling the light powder causes it to fly in the air to such an extent that there is substantial loss of material, and an annoyance to workmen handling the material and breathing the dust.

Numerous attempts have been made to overcome these objectionable properties of carbon black and render it denser and less dusty, but all such attempts heretofore have greatly changed the very essential physical properties of the carbon black which make it industrially valuable. Thus, the carbon black may be made into a paste with water or other volatile liquid such as gasoline, solvent naphtha, xylene, turpentine, carbon tetrachloride and the like, and the volatile liquid then distilled or evaporated, but the resulting cake is hard, the carbon particles tend to hold together in chunks, and they no longer disperse readily and uniformly as for example in rubber compositions. Apparently the state of aggregation of the carbon particles has been changed in such altered carbon blacks, and, as a matter of fact, the industrially essential physical properties, namely, easy and uniform dispersion, are so much impaired that such processes have never been a commercial success.

We find that when the original carbon black is added to a liquid to form a paste and the liquid slowly distilled or evaporated, such treatment causes changes which are associated with a free meniscus, i. e. vortices, surface tension adhesion, settling, volume contraction of the black, and a very persistent adhesion of particle to particle. The result is the clumping together of the carbon and the loss of dispersibility, as referred to above. This change of colloidal properties or loss of dispersibility may be mitigated or avoided by drying very quickly with violent agitation, drying by quickly removing the liquid by absorbing it into the walls of a small porous container, or by displacing the original liquid by another in such a manner as to avoid free suspension of the particles, in a liquid phase.

The main object of our invention is to secure carbon black having the desired dispersive properties, greater density, freedom from dust, freedom from gritty particles, less absorbed or occluded gases, reduced oil absorption than the ordinary powder form, and capable of considerable handling without crushing or dusting.

We have discovered that we can improve carbon black as to all or substantially all of the above mentioned properties without deleteriously affecting any of the properties essential to its satisfactory industrial use, such as softness and dispersive qualities, and by a process which includes the simultaneous or successive treatment of the carbon black with two immiscible liquids, the second liquid having greater attraction for or greater ability to wet the particles.

This process, if carried out under certain conditions, causes the carbon black to form into pellets which are hard enough to stand any ordinary shipment or handling without dusting, flying or breaking down, and which at the same time are easily crushed by moderate pressure, as between the fingers or by the pressures commonly employed in the rolls of rubber compounding machinery, printers ink mixers and the like. The crushed particles have substantially their original softness and the material disperses freely without leaving any particles of undispersed carbon in the material.

While the pellet form is a very convenient form of the carbon black, the shape of the particles is not the most important characteristic of this novel carbon black. The properties enumerated above may be incorporated into the black in the form of flakes

or the pellets or flakes may be crushed and pressed into blocks without seriously affecting its valuable dispersive properties.

In carrying out the process we mix the carbon black with one liquid and add a second liquid which is substantially immiscible with the first and which has greater ability to wet the carbon particles. The mixture is then agitated until the first mentioned liquid is practically free from carbon. This liquid is then separated and the other liquid is removed by evaporation.

Merely as an example of our improved process, the operation may be carried out as follows:

The carbon black is first mixed with water so as to form a comparatively thin paste with most of the occluded gases driven out. After thorough mixing gasoline is added and the whole mass thoroughly agitated. As a result substantially all of the carbon black floats on top of the water in the form of pellets and may be readily removed by filtering, decanting or the like. These pellets may then be dried to remove the absorbed gasoline and such moisture as there may be contained therein. Care should be taken to insure the complete wetting of the particles with the water, as otherwise the particles will be wet directly by the gasoline and in drying harsh particles may result. In the drying operation the carbon black retains its globular, ball-like or pellet form.

On the first addition of gasoline to the black and water paste, with agitation, the mass seems to thicken like an emulsion; then on further shaking, it seems to flocculate into granules; as the agitation goes on, a slight creaming to the top of the pellets or a scum takes place, then gradually the pellets form and float to the top. The end point is reached when the water below is free of carbon-clear.

The pellets are then screened off by any of the various methods, placed in an oven or a recovery apparatus and freed from solvent and water. It has been found that by use of this method the drying has little or no harmful effect upon the particle. Low temperature drying, and high temperature drying (around 400° Fahrenheit) leave the particle in a good soft condition. There are however various factors which will influence this.

If there is no organic residue decomposed by heat, there will be no binding of the particle. If there is, a harshness will result due to the decomposition or gumming of the residue. If too much gasoline is used, the particles will compact to comparatively hard pellets. If there is too much water and not enough gasoline, an inferior product will result.

The control however of the above process does not necessarily have to be delicate. A rather wide margin of safety exists.

Merely as an example of the relative proportions which we have found to operate satisfactorily, we give the following by weight:

	Parts
Carbon	5
Water	40 to 80
Gasoline	7 to 9

In carrying out our improved process with these proportions, the mass may be thoroughly shaken or otherwise agitated for about five minutes and the resulting pellets will be of about one-sixteenth of an inch in diameter and will be fairly uniform in size.

The pellets are very porous, of substantially spherical or globular form, have a smooth somewhat lustrous outer surface which is not easily broken by handling, are more compact than untreated carbon, are fragile under light pressure, and may be easily reduced to soft minute particles which cannot be told from the original particles except that possibly they have a more unctuous feel. They somewhat resemble lead shot and may be rolled in the hand without dirtying or dusting. Apparently the outer surface portion or shell of each pellet is slightly more compact than the inner part, but still porous.

In shipping or storing, we find that approximately twice the number of pounds of these pellets can be placed in a container of a given size than is the case with the untreated carbon black. Thus, expense is reduced for shipment or storage.

It will be noted that during the agitation and separation any grit which there may be in the carbon black may settle to the bottom, while the pellets float to the top and thus the purification of the carbon black will be effected at the same time the pellets are formed.

The pellets will not pack down in the bottom of a bag or other shipping container as in the case with untreated black and may be stored in bins and delivered through chutes to the point of use, without caking or sticking. The non-flying properties permit better control of the mixing in rubber or other composition, facilitate weighing, result in a substantial saving of material by eliminating mechanical loss, and add to the comfort of the operator.

The process of the present invention when carried out as described above yields substantially pure carbon black, but we find that the present invention can be employed for the manufacture of carbon black containing other materials uniformly distributed through or absorbed by the black, without loss of dispersibility. For example in its application in the rubber industry, it has been attempted to incorporate various other substances with the black, such as accelerators, softeners and anti-oxidants. When this is done by adding the said additional substance and the carbon black to a liquid in which the added ingre-

dient is soluble, and subsequently evaporating or distilling the liquid, the resulting carbon black is hard and has lost its ready dispersibility, as noted above. We find that the addition of substantial proportions of certain other substances to either of the liquids used in the process of the present invention does not impair the dispersibility of the resulting carbon black. Thus for example hexamethylene tetramine and various water soluble xanthates may be added to the water containing the carbon black or such substances as diphenyl-guanidine, thiocarbanilide, various amine oleates or stearates, or anti-oxidants, such as hydroquinone, resorcinol or naphthylamine may be added to the gasoline, benzol or water insoluble phase up to the limit of their respective solubilities, without impairing the qualities of the resulting carbon black.

There are various factors which enter into the process and these may be varied to get the pellets harder or softer or larger or smaller. Among these factors are the thickness of the paste, the amount of gasoline used, the adding of the gasoline in bulk or a little at a time, speed of agitation, temperature, type of gasoline used, and character of the carbon black. In using gasoline, it is desirable to use a high grade, such for instance that commonly known as aviation gasoline, in which there is a minimum amount of high boiling constituents. Other liquids, such as benzol, chloroform, toluol, carbon tetrachloride, xylol, turpentine, or carbon di-sulphide, may be used. It is important that the liquid used be one which is as nearly completely immiscible with water as possible.

Using an agent, such as carbon tetrachloride, chloroform, carbon di-sulphide, or any other of like character having a specific gravity of over one, will not cause the flotation of the pellets to the top of the water, but they will simply sink to the bottom. They can however be recovered by filtration or screening.

Some creaming agents have greater wetting power than gasoline, and therefore require a lesser amount to bring about the end point.

In general it is preferable that the creaming agent be completely volatile so as to leave the carbon pure. In special cases it is not necessary where the residue will impart special properties to the pellet.

If small pellets are desired, a lesser amount of gasoline or other liquid should be used in respect to the amount of water and carbon, and greater agitation should be employed. To secure large pellets, we use a larger amount of gasoline and slower agitation. In practice, we do not consider a size larger than one-quarter of an inch desirable. There are many kinds, grades or varieties of carbon black and often identification of the particular kind or grade is difficult. With our improved process the different kinds or grades may be made into pellets of different sizes so that identification is facilitated, for instance, very small pellets may be made for printers ink and larger ones for rubber, etc.

It is possible also to reduce the flying of the black by not carrying the above treatment to its ultimate pellet stage. It will come out as an amorphous mass somewhat flaky and fluffy, and must be separated from the mixture by filtration.

The flake form of the material is produced by incorporating the black in water, as described above, and immersing in the mixture a porous material which is wet with gasoline, or other liquid as before described. Owing to the preferential wetting power of the gasoline, the carbon particles leave the water and are wetted by the gasoline, building up a layer instead of forming globules or pellets as is the case when the agitation method is used. The surface on which the black is deposited may be a rotating porous cylinder, with the gasoline supplied to the inside, the layer of black being continuously scraped off the cylinder, as it rotates.

Another method of producing the flake form is to crush the pellets before evaporating the solvent, as between rolls, and then evaporating the gasoline, or other volatile liquid.

The whole process of pellet formation depends upon the selective wetting power which the various solvents exert on the carbon black.

We use the term "carbon black" in its broad rather than limited meaning, and by such term include lamp black, gas black and other black analogous carbon substances for use as pigments or fillers.

We find that the present invention can be applied to other fine powders whose industrial applications depend upon their dispersibility. For example, precipitated magnesium carbonate, zinc oxide, titanium oxide, barium sulfate, and certain talcs and clays are difficult to disperse in rubber or oil on account of absorbed air and tendency to fly as dust, and we find that these powders can be manufactured in denser form, with elimination of most of the absorbed air and without loss of dispersibility, by the process of the present invention.

We have obtained very satisfactory pellets from magnesium carbonate and also from zinc oxide by use of the process exactly as described above although benzol has been used instead of gasoline.

In the foregoing we have referred to agitation of the mixture, but we do not wish to be restricted to any particular kind of agitating apparatus. For batch operation the agitation may be accomplished by violent shaking, rotating or tumbling of the container, and for continuous operation the agitation

may be by any means which will impart rapid changes in the direction and/or velocity of the flow of the mixture stream, as for instance, apertured obstructions or baffles in a conduit.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. Substantially pure carbon black in the form of commercially uniform, comparatively small, rounded, smooth aggregates having a spongy porous interior.

2. As an article of manufacture, a pellet of approximately one-sixteenth of an inch in diameter and formed of a porous mass of substantially pure carbon black.

3. A carbon black pellet formed of soft particles, the outer surface of the pellet being smooth and the outer portion being slightly denser than the inner portion.

4. The process of treating carbon black which includes the steps of mixing the carbon black with a liquid to form a very thin paste, adding a second liquid in smaller amount and which is immiscible with the first liquid, agitating, separating the carbon black and second liquid from the first liquid, and then separating the carbon black and the second liquid.

5. The method which includes wetting carbon black with water, bringing the mixture into contact with a liquid having greater wetting properties whereby the carbon black is substantially separated from the water, and evaporating the second liquid from the carbon black to leave aggregates of porous fragile structure readily reducible to the original state of fine subdivision.

6. A process of treating carbon black which consists in agitating it in the presence of two immiscible liquids and having dissolved or incorporated in one or the other of said liquids, accelerators, softeners, anti-oxidants or other useful ingredients, whereby the carbon black forms pellets in or with one of said liquids absorbing during this process, the accelerators, softeners, anti-oxidants or other useful ingredient from one or the other of said liquids.

7. The process which includes wetting carbon black with water, bringing it and gasoline into contact under conditions causing the gasoline to displace substantially all of the water from the carbon black, removing the carbon black mass wetted with the gasoline, and evaporating the gasoline.

8. The following process, mixing 40 to 80 parts of water and 5 parts of carbon black to form a thin paste, adding 7 to 9 parts of gasoline, agitating until the carbon black forms in pellets in the gasoline and leaves the water substantially clear, separating the pellets and the water, and drying the pellets.

9. The process of treating compounding materials in a fine state of subdivision to bring them into a more compact form for use in plastic and coating composition which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which it does not react chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the material from the first liquid, and drying to obtain the material in a more compact form.

10. The process of treating carbon black, which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which it does not coact chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the carbon black from the first liquid and drying to obtain the carbon black in more compact form.

Signed at New York, in the county of New York and State of New York, this 30 day of November 1927.

WILLIAM B. WIEGAND.
LOUIS J. VENUTO.

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CERTIFICATE OF CORRECTION.

Patent No. 1,889,429. November 29, 1932.

WILLIAM B. WIEGAND, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, line 12, claim 1, after "spongy" insert the word "or"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 21st day of February, A. D. 1933.

M. J. Moore,
Acting Commissioner of Patents.

(Seal)

[fol. 254] Patents Subsequent to the Patent in Suit Containing Tributes to Pelleted Carbon Black:

Cabot	2,120,540
	2,120,541
	Re. 19,750
	2,134,950
	2,039,766
Columbian	Re. 21,379
United	Re. 21,390
Huber	2,127,137
Atlas	2,131,686

234

PART OF PLAINTIFF'S EXHIBIT 4

255

June 14, 1938. E. BILLINGS ET AL 2,120,540

MANUFACTURE OF CARBON BLACK

Filed July 18, 1932

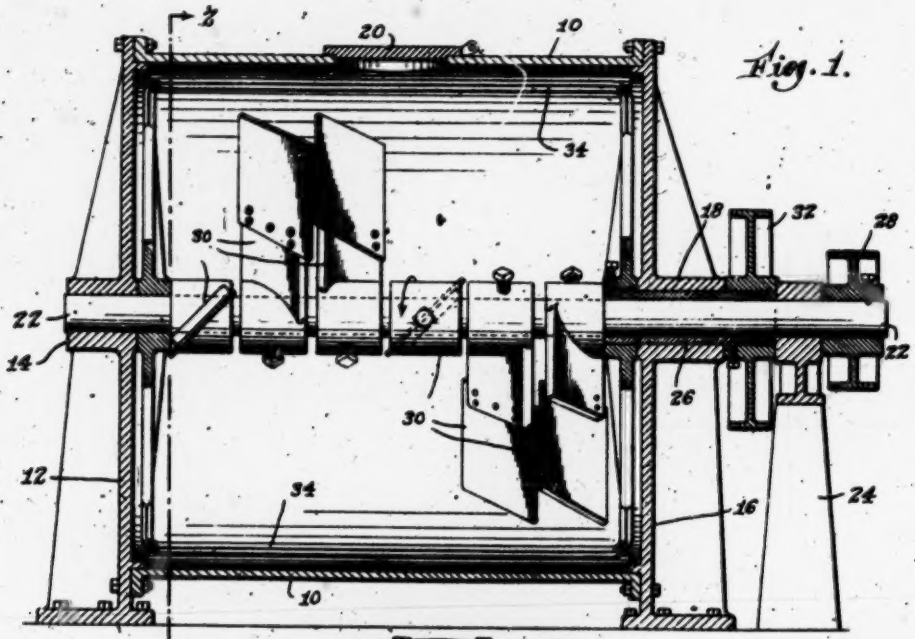


Fig. 1.

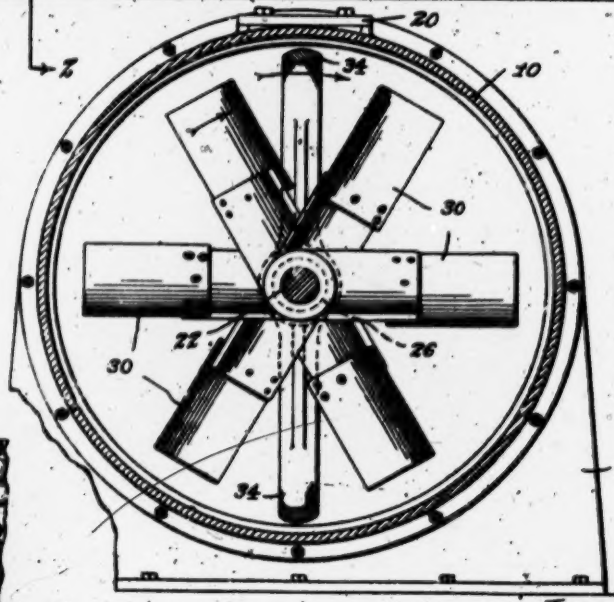


Fig. 2.



Fig. 3.

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Patented June 14, 1938

2,120,540

UNITED STATES PATENT OFFICE

2,120,540

MANUFACTURE OF CARBON BLACK

Edmund Billings, Weston, and Harold H. Offutt, Winchester, Mass., assignors to Godfrey L. Cabot, Inc., Boston, Mass., a corporation of Massachusetts

Application July 13, 1933, Serial No. 680,134

4 Claims. (Cl. 134—69)

This invention relates to improvements in the manufacture of carbon black and more particularly to a novel process of manufacturing from flocculent carbon black a new product consisting of carbon black in the form of granules of relatively high apparent density and of sufficient cohesion to withstand handling in bulk. The granules may be made substantially spherical in shape and the surfaces thereof may, if desired, be polished.

In the manufacture of carbon black as it is at present carried out by one commercial method, the black as removed from the channel surfaces, bolted to remove grit and delivered to settling or agitating tanks, has an apparent density of between 3 and 7 pounds per cubic foot. The carbon black so delivered is stirred slowly by rotating blades, consisting of rods or flat shapes extending radially from a shaft revolved at a rate of from 4 to 20 R. P. M. This gentle agitation results in a slow settling of the very light carbon black particles and a freeing of the mass from entrained air. The viscosity of the mass increases as settling proceeds, and in practice it has been found necessary to withdraw or pack off the black from the agitating tank before its density exceeds 15 pounds per cubic foot.

After the black has been removed from the agitating tanks, it is packed under pressure in bags or sacks. In this step it is customary to use a screw conveyor or feeder rotating about a vertical axis and arranged to force itself up out of the sack as the black is delivered and forced into the same. The resulting package is of a density termed "uncompressed" in the trade and does not usually exceed 15 pounds per cubic foot in apparent density.

A further increase in the density of the black is usually obtained by briquetting the packed sacks in powerful hydraulic or mechanical presses, and in this way a maximum apparent density of 27 to 30 pounds per cubic foot is attained.

The steps above outlined are necessary in order to bring the light flocculent carbon black into a condition in which it can be handled economically and conveniently in packaging for distribution, as well as into convenient shape for the consumer to use. For example, the light fragile particles of carbon black as it is first produced not only occupy enormous volume in mass but are likely to be scattered and diffused by every breath of air, and probably repel each other on account of static electrical conditions to which they are subject.

The present invention rests upon the discovery that by subjecting a mass of carbon black particles, in a dry state, to multi-directional pressure the carbon black is converted into granules which are relatively tenacious in structure, relatively non-coherent and non-adherent of surface and of an apparent density in bulk of more than 20 pounds per cubic foot. This conversion of flocculent carbon black to granular form is effected without the use of any binding ingredient whatever and the agglomeration of the particles is, so far as we understand the phenomenon, by reason of impact-generated cohesion of the carbon black itself. The novel product thus produced, which may preferably be of substantially spherical granular shape, possesses striking commercial advantages. In the first place, it is substantially dustless in that most of its particles are agglomerated into granules of appreciable size and substantial density. In the second place, the non-adherent character of the surfaces of the granules reduces the apparent viscosity of the mass, which viscosity may be further reduced by the preferred spherical form of the granules, permitting the carbon black to be conveyed through pipes by gravity or by streams of air, to be transported in tank cars and to be stored in bulk. Besides being much more cleanly for the consumer to handle, the novel product when mixed with other ingredients disperses and diffuses more completely and perfectly in various compounded materials than commercial carbon black of forms heretofore known.

The precise steps leading to the production of this new product of manufacture may be varied to some extent so long as each nucleus or center is subjected to impact or pressure from substantially every direction. One manner of securing satisfactory condition is to maintain the carbon black for a sufficient interval of time in a state approaching perfect turbulence, and by this we mean that each particle has such freedom of motion and is associated with other particles in sufficient number to insure the multi-directional pressures or impacts required. For example, a charge of commercial carbon black may be converted into the desired granular form by being properly agitated in a drum so that the requisite pressures result from the impact of the agitating blades, the impact of one particle against another and the impact of the particles against the walls of the drum as we understand the phenomena.

Another manner in which the desired results may be secured is by treating a charge of commercial carbon black in a ball mill of the horizontal drum type. In this case, the requisite

pressures may be derived principally from the rolling impact of the particles as they drop from particle to particle in rolling down the surface of the shifting mass in the mill.

In whatever apparatus the carbon black is treated, the process of conversion to the desired granular form is expedited by including in the mass a quantity of carbon black in its final form, that is to say, a mother or priming charge in granular form. The explanation of this probably is that an increased number of pressure supplying units is thereby provided so that the agglomeration, shaping and compacting of the individual dense grains takes place more rapidly than under conditions wherein the balling of the carbon black is brought about in an amorphous mass. The introduction into the process of this seeding or priming step is advantageous principally because it reduces the time required for effecting a complete agglomeration or conversion of the charge but it is not otherwise essential.

The nature of the invention will be best understood and appreciated from the following description of one form of apparatus which is well adapted for the production of granular carbon black in a substantially spherical form, together with an explanation of its manner of use. In the accompanying drawing,

Fig. 1 is a view in longitudinal section of such apparatus;

Fig. 2 is a view in cross section of the apparatus on the line 2—2 of Fig. 1, and

Fig. 3 represents carbon black of a preferred spherical form as seen in the field of a microscope, magnified about 50 diameters.

The apparatus comprises a horisontally disposed tank or drum 10 which is formed from a cylindrical shell 10, flanged and bolted to end plates 12 and 16. The end plates are both flanged to provide a firm footing and have outwardly-extending bosses 14 and 18 respectively in which are formed bearings for concentric driven shafts 22 and 26. The inner solid shaft 22 is journaled in the boss 14 of the left-hand end plate 12 and in bearings formed in an outboard standard 24. It is provided at its outer end with a driving pulley 28 and carries within the drum a series of blades 30. As herein shown, six blades are provided spaced longitudinally of the shaft and disposed 60° apart and inclined alternately substantially 45° to their axis of rotation. The blades 30 do not extend to the inner wall of the drum 10 but terminate so as to leave a substantial clearance. For example, in a 14 inch drum the clearance between the ends of the blades and the inner wall of the drum may advantageously be as much as three-quarters of an inch. The provision of an ample clearance is of importance because it prevents the blades from sweeping through carbon black adhering to the inner walls of the drum. Such treatment of the carbon black tends to shear the spherical granules being formed and to form sheared surfaces which are sticky and so inimical to the formation of the preferred spherical grain form of carbon black. The exact arrangement and shape of the blades 30 is of secondary importance. As herein shown, they are inclined alternately in opposite directions but any arrangement of the blades which will produce a turbulent stirring and agitation of the dry carbon black contained in the drum is satisfactory.

At its right-hand end the shaft 22 passes through a short hollow shaft 26 secured at its outer end to a driving pulley 32. Within the drum 10 the inner end of the hollow shaft 26 carries a ball-shaped agitator or scraper 34. This is journaled at its left end upon the shaft 22 and is disposed to rotate slowly in the space between the ends of the blades 30 and the inner wall of the drum 10. The hollow shaft and the scraper 34 are driven at a slow rate by the pulley 32 in the same direction of rotation as the blades 30. It will be noted that a substantial clearance is provided between the scraper 34 and the wall of the drum 10. The principal function of the scraper 34 is to remove from the walls of the drum the carbon black which is built up thereon in the form of a coating and which would otherwise become thick enough to extend within the path of the blades 30. By this arrangement, shearing of the grains by the blades 30 is substantially eliminated and the total amount of shearing in the apparatus is minimised on account of the slow movement of the scraper 34.

In operating the apparatus herein disclosed, the drum 10 may be filled to about one-half its capacity with commercial flocculent carbon black or, as already intimated, a mixture of commercial carbon black with a small quantity of spherical grain carbon black. The charge may be introduced and withdrawn through a hand hole provided in the upper surface of the drum 10 and closed by a cover 20. The two shafts are then set in operation. In a small apparatus, where a 14-inch drum is employed, a speed of approximately 440 R. P. M. for the shaft 22 and the blades 30 has been found suitable for imparting a turbulent agitation to the black contained in the drum. At the same time, the scraper 34 may be rotated at a speed of 16 to 18 R. P. M. in the same direction. The result is that the charge of carbon black is stirred or churned into a state of turbulent agitation, in which the particles are subjected to multi-directional pressure by impact with other particles and by impact against the blades 30 and scraper 34, and impact against the walls of the drum and, under these conditions, there occurs a systematic and progressive agglomeration of the particles upon each other, or upon the granules of the priming charge, and a shaping of the agglomerates into spherical granules having hammered or impact-formed surfaces which present the striking characteristics of dustlessness, high density and non-adherent, free flowing contact with each other. In general, it may be stated that the apparent density of the carbon black granules is quickly brought to approximately 30 pounds per cubic foot. At this density the granules are 200 to 250 mesh in diameter and may be increased in size by the addition of more flocculent carbon black as the process is continued.

The general appearance of a preferred form of the finished product, as seen in the field of a microscope, is represented in Fig. 3. It will be noted that in this particular form the granules may be spherical, or approximately so, and that there is a substantially uniform upper limit of diameter which has been attained by a large number of the granules.

In addition to the characteristics of relatively high density and non-coherent surface, the carbon black granules produced as above outlined are relatively tenacious in their structure. They cannot be crushed between the fingers by normal pressure, although they may be readily crushed against a hard surface or by rubbing which subjects them to shearing stress between the fingers.

The process of our invention may be success-

fully carried out at moderate temperature. The sticking properties of sheared particles of carbon black tend to disappear as the temperature is increased, but since carbon black will react rapidly with oxygen in the neighborhood of 500° F. it is important to carry out the process well on the safe side of that temperature, since any oxidising effect in the carbon black alters and impairs those characteristics of the material which are particularly desired in the rubber and printing ink industries. Accordingly, while it may be possible to carry out successfully the process of my invention at a high temperature by taking steps to exclude oxygen from the carbon black, it is preferable to carry out the process below a temperature at which there is tendency to rapid oxidation. For example, in using the illustrated apparatus to convert a charge of commercial carbon black which has not been primed by the addition of spherical-grain carbon black, a temperature of about 400° F. has been found satisfactory. For this purpose the walls of the drum may be heated by any desired or convenient means.

While we have explained to some extent a theory of the formation of the dense, preferably spherical granules which it is the object of our invention to produce, there may be other important factors in the phenomenon of its formation which are not at the present time fully understood. It is undoubtedly true that agglomeration of the particles is retarded by the cushion of air between them at the instant of impact, and that this unfavorable factor is overcome to some extent by imparting a substantial velocity to the particles so that sufficient momentum and pressure of contact are developed to squeeze out the interposed air at their points of contact.

It is, further, probable that the behaviour of the carbon black particles is affected by charges of static electricity carried by them. We are unable to state, from the electrical standpoint, just what is the action of the carbon black particles and how the formation of spherical-grain carbon black is affected by electrical conditions.

Another factor which requires attention is the pressure to which the carbon black charge is subjected during the process of its agglomeration. It is desirable to limit the depth of the charge so that the particles at the bottom of the mass shall not be subjected to too great weight and that the blades of the agitating mechanism shall not encounter too great resistance in creating the desired turbulent agitation of the particles. In the illustrative apparatus, a depth of 7 to 8 inches results in satisfactory pressure conditions. The foregoing disclosure, however, is adequate and complete from the practical standpoint and sufficient to enable one skilled in the art to produce from commercial flocculent carbon black the novel product of manufacture of our invention.

It will be understood that the apparatus herein illustrated and described constitutes only one example of apparatus which we have found suitable for the processes of our invention. Sandy or granular carbon black may be produced from the flocculent carbon black of commerce by employing a ball mill, wherein the mass of carbon black is maintained in a continuous condition of surface flow, with the result that the particles are poured or rolled over each other and caused to agglomerate and take granular form by reason of the rolling contact thus imparted to them. The carbon black particles are so small that the rolling impact to which any point thereof is subjected becomes very appreciable expressed in pounds per square inch of pressure, and the hammered, impact-formed, non-coherent surface desired is produced in this manner and may become polished if continued long enough.

We have found, while operating a large scale apparatus, that the degree of turbulence created is of prime importance because too great a degree of turbulence, that is to say, too rapid motion of one particle with respect to its neighbors, especially when all are subjected to pressure due to the weight of other particles above them or to centrifugal pressure or to other mechanical or gravitational causes, tends to create a shearing effect between particles which is quite as detrimental to the successful carrying out of the process as is the shearing effect between the particles on the rapidly moving blades or the stationary walls of the apparatus. It should be noted, therefore, that the differential speed between the particles of carbon black within adjacent zones of the apparatus must not be excessive. We have found that by careful attention to these factors it is possible to eliminate a scraper bar corresponding to that shown in the illustrated apparatus, that is, that at a certain clearance between the moving blades and the stationary wall and at a certain speed of revolution, the differential speeds of the carbon black particles will be such as practically to eliminate the objectionable shearing effect. While the scraper may be eliminated under these conditions, it is a desirable and convenient safeguard for commercial operation.

In referring to the multi-directional pressures to which the carbon black particles are subjected, it appears probable that such impacts or pressures should not occur twice in succession in the same direction. The novel product herein disclosed is not herein claimed but forms the subject matter of our application Serial No. 684,884, filed August 12, 1933.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A process of producing carbon black in the form of granules of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in confining a mass of flocculent carbon black of commerce in a horizontal drum, subjecting it to turbulent agitation by passing rotary blades through the mass and simultaneously scraping the sides of the container to remove wall cake therefrom and continuing such agitation until the carbon black agglomerates into granules which are self-sustaining but capable of disintegration under shearing pressure.

2. A process of transforming flocculent carbon black into a substantially dustless mass of free flowing, relatively tenacious, self-sustaining granules, transportable in bulk without disintegration, which consists in subjecting such carbon black in a dry state to turbulent agitation by rotating at substantial speed a series of relatively broad blades through a mass of said carbon black in a closed drum, said blades being in staggered relation, with the surface of revolution thereof adjacent to and substantially uniformly spaced from the wall of the drum, whereby the entire mass of said black within the container is maintained in a state of substantially perfect turbulence, and continuing such turbulent agitation until said black assumes the form of granules as above defined.

3. A process of transforming flocculent carbon black into a substantially dustless mass of free flowing, relatively tenacious, self-sustaining granules, transportable in bulk without disintegration, which consists in subjecting particles of such carbon black in a dry state to multi-directional impact with other carbon black particles by turbulent agitation thereof by rotating at substantial speed through a mass of said carbon black in a closed drum a series of relatively broad blades with faces of successive blades inclined in opposite directions in relation to the axis of rotation and with the surface of revolution thereof adjacent to and substantially uniformly spaced from the wall of the drum, whereby the entire mass of said carbon black is maintained in a state approaching perfect turbulence, and continuing such turbulent agitation until said carbon black is converted into granules as above defined.

4. A process of treating flocculent carbon black, which consists in maintaining said carbon black in a state approaching perfect turbulence by rotating a series of relatively broad blades at substantial speed through a mass of said carbon black in a drum, the faces of alternate blades being at different angles to the axis of rotation, and thereby subjecting all parts of said mass to turbulent agitation, and simultaneously scraping and maintaining free from wall cake the wall of said drum, all as and for the purpose described.

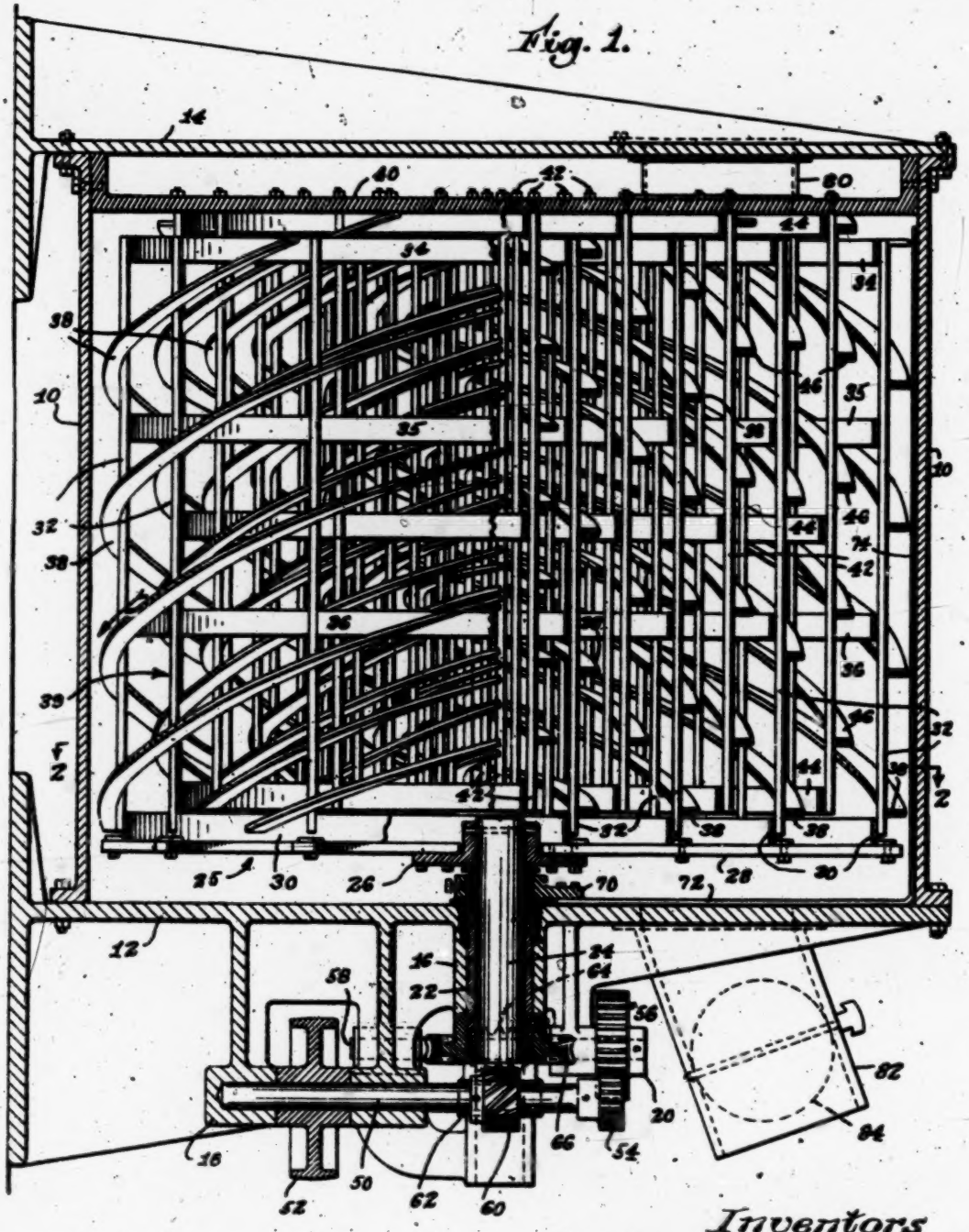
EDMUND BILLINGS.
HAROLD H. OFFUTT.

June 14, 1938. E. BILLINGS ET AL 2,120,541

MANUFACTURE OF CARBON BLACK

Filed Nov. 16, 1932 2 Sheets-Sheet 1

Fig. 1.



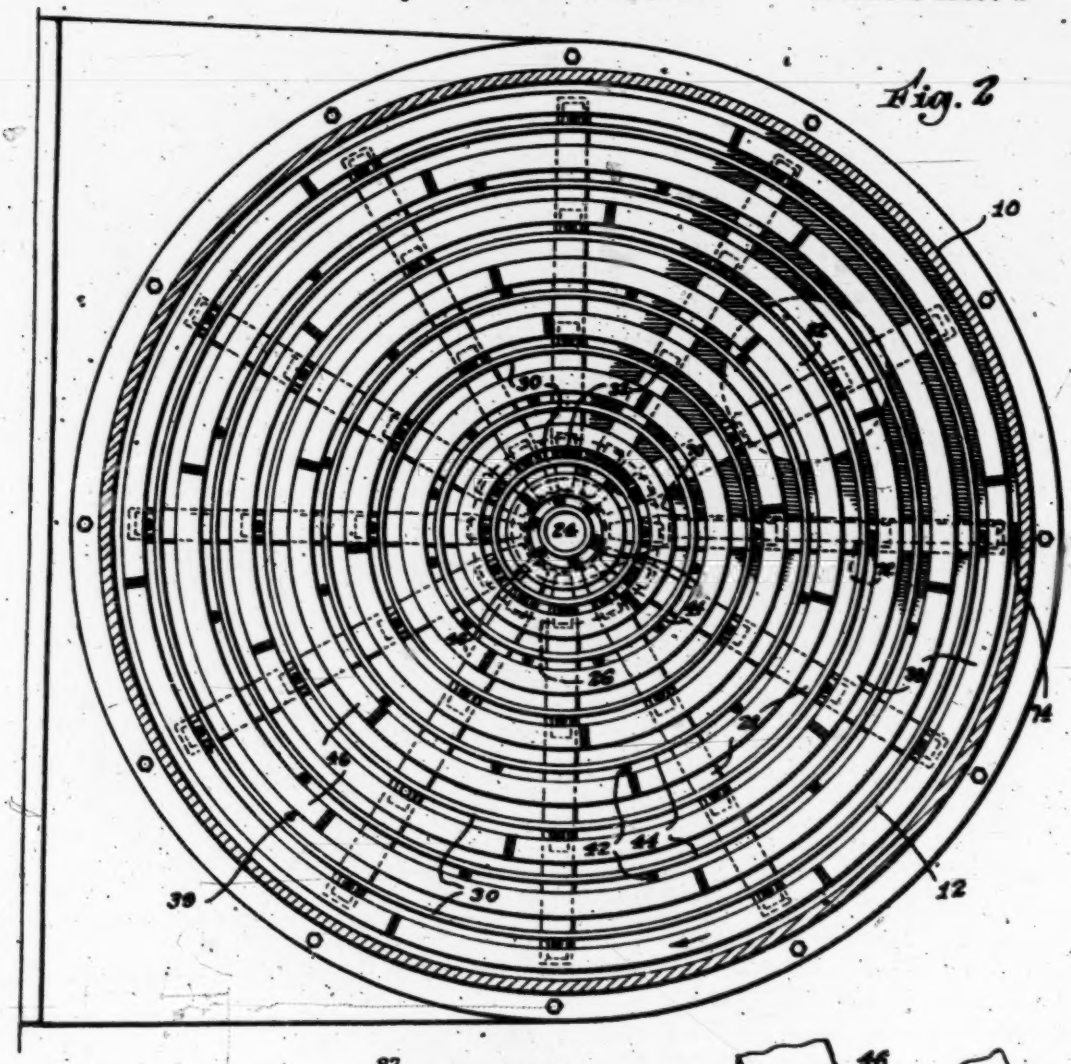
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MANUFACTURE OF CARBON BLACK

Filed Nov. 16, 1932 2 Sheets-Sheet 2

Fig. 2



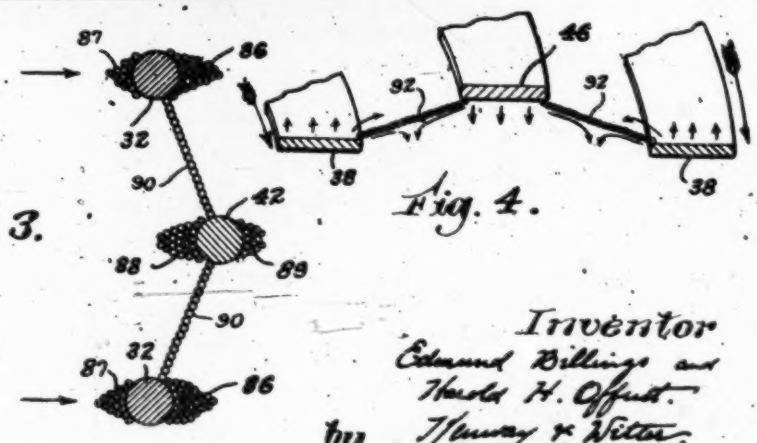


Fig. 3.

Fig. 4.

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Patented June 14, 1938 262 2,120,541

UNITED STATES PATENT OFFICE

2,120,541

MANUFACTURE OF CARBON BLACK

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Application November 16, 1932, Serial No. 642,850

29 Claims. (Cl. 134—60)

This invention relates to improvements in the manufacture of carbon black. In one aspect it consists in improvements in the process disclosed in our copending application Serial No. 623,184, filed July 18, 1932, for manufacturing carbon black in the form of granules of relatively high apparent density. The present application is a continuation in part of that application as to all common subject matter.

This invention, as well as that of our said copending application, rests upon the discovery that by subjecting carbon black particles in a dry state to multi-directional pressure, at the same time maintaining them in a state of turbulence, they are caused to agglomerate with each other and to associate together in a novel form, viz., the particles are built up systematically and progressively into spherical granules, relatively tenacious in structure, relatively non-coherent and non-adherent of surface and of an apparent density of more than 18 pounds per cubic foot. The novel product thus produced possesses striking commercial advantages. In the first place, it is substantially dustless in that nearly all of its particles are agglomerated into granules of substantial size and density. In the second place, the density and the non-adherent character of the surface of the granules reduces the apparent viscosity of the mass and permits the carbon black to be conveyed through pipes by gravity or by streams of air, to be transported in tank cars and to be stored in bulk. Beside being much more cleanly for the consumer to handle, the novel product when properly mixed with ingredients such as rubber disperses as completely and perfectly as commercial carbon black of forms heretofore known.

The novel product above described is the subject matter of U. S. Letters Patent No. 1,967,314, granted May 1, 1934 upon our copending application. The process herein disclosed comprises one of several whereby that product may be manufactured economically on a commercial basis.

The precise steps leading to the production of this new product of manufacture may be varied to some extent so long as each nucleus or center is subjected to impact or pressure from substantially every direction. One manner of securing satisfactory conditions is to maintain the carbon black for a sufficient time in a state approaching perfect turbulence under conditions of turbulent pressure such that each particle has freedom of motion and is associated with other particles in sufficient number to insure the multi-directional pressures or impacts required. For example, a charge of commercial carbon black may be converted into the form of relatively hard, dense and spherical grains by being properly agitated in a drum so that the requisite pressures result from the impact of the agitating blades, the impact of one particle against another and the impact of the particles against the walls of the drum.

Another manner in which the desired results may be secured is by treating a charge of commercial carbon black in a mill of the character of a ball mill. In this case, the requisite pressures may be derived principally from the rolling impact of the particles as they drop from particle to particle in rolling down the surface of the shifting mass in the mill or from the impact of particles against the rotating walls.

In whatever apparatus the carbon black is treated, the process of conversion to the dense granular form is expedited by including in the mass a quantity of carbon black in its final form, that is to say, a mother or priming charge in said granular form. The explanation of this probably is that an increased number of pressure and impact supplying units is thereby provided and the requisite degree of turbulence facilitated so that the agglomeration, shaping and compacting of the individual dense spherical grains takes place more rapidly than under conditions wherein the building up of the carbon black granules is brought about in an amorphous mass. The introduction into the process of this seeding or priming step is advantageous principally because it reduces the time required for effecting a complete agglomeration or conversion of the charge but it is not otherwise essential.

Commercial carbon black in bulk is very viscous and for this reason offers considerable resistance to the turbulent motion above described, especially when treated in relatively large bulk. We have discovered that the speed of the process is somewhat enhanced by intensifying the impact nature of this turbulence and that such improvement may be effectively brought about by subjecting the carbon black to turbulent pressure between opposing surfaces relatively movable toward and past each other to agitate and impact the black therebetween in a turbulent manner. This action brings the particles comprising the black into a most intimate and bombarding relation, whereby they are more speedily and completely compacted into the relatively hard and dense form above described. The efficiency of the process and the consistency and

value of the product also depend to a very large extent upon the uniformity of the treatment of all the parts of the carbon black during this operation and for this reason we also agitate or move the black transversely of and simultaneously with the turbulent movement imparted as above explained. This latter agitation serves to circulate the bulk of black continuously during the treatment and thereby tends to keep the product uniform at all times. We also prefer to oppose somewhat this latter agitation, whereby such movement serves not only to keep the product uniform but also aids in the impacting of the black into its final dense form. Thus the black is agitated in two directions at an angle to each other and, while being kept of a uniform consistency, is impacted into dense form by the opposition to its movement in said directions and the impacts and encounters.

The nature of the invention will be best understood and appreciated from the following description of one form of apparatus which is well adapted for the production of spherical grain carbon black in accordance with our improved process. In the accompanying drawings,

Fig. 1 is a vertical sectional view through an apparatus embodying our invention;

Fig. 2 is a plan view partly in section on line 2—2 of Fig. 1;

Fig. 3 is a fragmentary view on an enlarged scale illustrating diagrammatically the horizontal movement of the carbon black particles in the apparatus; and

Fig. 4 is a fragmentary view in vertical section illustrating diagrammatically the vertical movement of the carbon black particles in the apparatus.

The apparatus comprises a stationary vertically-disposed tank or drum 10 which is formed from a cylindrical shell flanged and bolted to bottom and top plates 12 and 14. Depending from the bottom plate is a boss providing a vertical bearing 16 and brackets providing horizontal bearings 18 and 20. Rotatably mounted in the vertical bearing 16 are a sleeve 22 and a shaft 24, the sleeve and shaft extending upwardly into the drum 10.

Mounted on the upper end of the shaft 24 and within the drum 10 is a rotatable cage 25 supported on a flanged collar 26 pinned to the shaft. This cage comprises a plurality of radially-extending bottom bars 28 supporting thereon four concentric bands 30 disposed edgewise in spaced relation. Carried by each band 30 and extending vertically upward therefrom are a plurality of impelling elements in the nature of rods 32 circular in cross section. The top ends and the intermediate portions of the rods are connected together by similar concentric bands 34, 35 and 36. Also carried by each circular row of rods 32 are one or more spirally arranged straps or blades 38.

An inverted cage 39, similar to the cage 25 supported on the collar 26 and just described, is mounted in stationary position on a plate 40 secured within the upper end of the drum 10. This stationary cage 39 comprises a plurality of circular rows of rods 42, corresponding to the rows of rods 32 and spaced concentrically, carried by and depending from the plate 40. The rods 42 in each row are connected together by three or more bands 44 and each row of rods also has mounted thereon one or more spirally-arranged straps or blades 46, the stationary straps 46 being pitched in the same direction as and arranged in a manner similar to the movable straps 38.

A relative rotary movement is provided between the two cages by rotating the shaft 24 on which is mounted the cage 25. A shaft 50, having a driving pulley 52 thereon, is mounted in the bearings 18 beneath the drum 10 and on the opposite end of this shaft is a gear 54 arranged in mesh with a relatively larger gear 56 on a parallel shaft 58 journaled in the bearings 20. A spiral gear 60 on the shaft 58 is arranged to mesh with a like gear 62 on the vertical shaft 24 and a worm 64 on the shaft 58 is in mesh with a worm gear 66 on the lower end of the sleeve 22. Through these connections the pulley 52 is connected to rotate the first described cage 25 in a counter-clockwise direction as shown by the arrow (Fig. 1) and to rotate the sleeve 22 in the same direction at a relatively slower speed.

The sleeve 16 is provided with a flange 70 to which is secured a scraper having a horizontal portion 72 extending radially outward along the bottom wall of the drum and a vertical portion 74 extending upwardly along the side wall of the drum, a substantial clearance being provided between both parts of the scraper and the drum walls. The scale of the drawings does not permit showing this clearance. The principal function of the scraper is to remove from the bottom and side walls of the drum the carbon black which would otherwise build up thereon in the form of a coating or shell and which would otherwise become thick enough to extend within the path of movement of the straps 38 and seriously vary the clearance between the side walls and the elements 32. It will be understood that to permit the product to build up on these walls would not only deleteriously affect the desired turbulent motion of the product, reduce the effective capacity of the apparatus and increase the power consumption unnecessarily, but would produce a shearing and breaking up of the treated product and thus to some extent defeat the purpose of the treatment. The sleeve rotates the scraper at a reduced speed which is sufficient to keep the walls free from any substantial accumulation of the black, and by this arrangement shearing of the grains by the rotary cage or the scraper is substantially eliminated and the total amount of shearing in the apparatus is limited to a minimum.

The charge of flocculent carbon black may be introduced into the drum through an opening provided through the top wall at 80 and the finished product may be drawn off through a discharge pipe 82 in the bottom plate 12, a damper valve 84 being provided within the discharge pipe.

In operating the apparatus herein disclosed, the drum 10 may be filled nearly to its capacity with commercial carbon black or, as already intimated, a mixture of commercial carbon black with a primary charge of spherical-grain carbon black. The shaft 50 is then driven to rotate the cage 25 in the direction of the arrow (Fig. 1), the scraper 72—74 rotating in the same direction at a greatly reduced speed. In an apparatus having a drum of 4-foot diameter, a cage speed of 45 R. P. M. and a scraper speed of 1½ R. P. M. have been found to be quite satisfactory. The result of this operation is that the charge of carbon black is stirred or churned into a state of turbulence in which the particles are subjected to multi-directional pressure by impact with other particles and by impact against the op-

posing and repelling surfaces and against the walls of the drum. Under these conditions there occurs an agglomeration of the particles resulting in a progressive and systematic building up of them into spherical granules having hammered or impact-formed surfaces which present the striking characteristics of high density and non-adherent, free-flowing contact with each other. In general, it may be stated that the apparent density of the carbon black may be brought to approximately 30 pounds per cubic foot. At this density the granules may be 250 mesh or larger in diameter.

In the light of our present knowledge, we may suggest one theory of what takes place within the drum to cause the carbon black to assume its new and relatively dense form by reference to Figs. 3 and 4 of the drawings. The stationary elements 42 are arranged alternately with the moving elements 32 and the charge of carbon black surrounds and entirely fills the space between these elements. As the elements 32 move relative to the elements 42 (Fig. 3), they tend to carry the black in a mass therewith but such movement of the black is opposed by the stationary elements 42. The result is that cones 96 and 97 of carbon black form both forwardly and rearwardly of the elements 32 and move along with these elements, while cones 98 and 99 of carbon black form both forwardly and rearwardly of the elements 42 remain stationary therewith. The carbon black intermediate the elements 32 and 42 (indicated by line 99 of particles in Fig. 3) is in a state of turbulence, the black adjacent to the elements 32 moving nearly as fast as those elements and the carbon black adjacent to the elements 42 remaining nearly stationary. The relative positions of the elements are constantly changing as the elements 32 approach and recede from the elements 42 and, due to this action, the particles of carbon black are alternately being brought into most intimate and bombarding relation with each other whereby they are compacted into the relatively dense form of the finished product and into a relatively loose relation, wherein they are free to rearrange themselves for the next impact. Thus the carbon black particles are intermittently subjected to multi-directional pressures or impacts and are relatively rearranged between the successive impacts, such impacts or pressures thereby not occurring twice in succession in the same direction.

Simultaneously with the above action, the spiral straps 38 are acting constantly to move portions of the carbon black mass upwardly and thereby keep the product uniform from bottom to top. It will be understood that since the pressure of the carbon black increases with its depth, the nature of its turbulent and bombarding action will correspondingly vary from top to bottom. By constantly moving the black from bottom to top, the entire charge is being continuously passed through the apparatus and thereby given a uniform treatment throughout. This upward movement of the carbon black by the stationary spiral straps 38 is, however, opposed by the spiral straps 46 which are secured to the elements 42. The result of this arrangement is somewhat illustrated in Fig. 4. Rotation of the spirals 38 tend to raise the carbon black upwardly; while centrifugal action may also somewhat move the black outwardly. That portion of the black located intermediate each two adjacent and concentric spirals 38 is intercepted by a spiral 46 which, in like manner, not only opposes such upward movement but tends to move the black downwardly, it being understood that in the apparatus illustrated, the mass of carbon black as a whole is also rotating in the drum though at a greatly reduced speed. When the shaft 34 and the cage 36 in this apparatus are rotating at 45 R. P. M., the mass of carbon black ordinarily rotates at about 4 R. P. M. Thus the spirals 38 and 46 not only serve to keep the product uniform from top to bottom but also set up an opposing motion of the carbon black in a substantially vertical direction, similar to the horizontal motion set up by the elements 32 and 42, which materially aids in developing the necessary pressure of impact of the black and its conversion into its final dense form. This movement and opposing action of the black are illustrated by the arrows and the lines 92 of black particles in Fig. 4.

When the treatment of a charge of carbon black has been continued in this manner from 60 to 120 minutes, it will be found that practically the entire charge has been converted from its original flocculent condition to the granular form above defined, in which all the grains are substantially spherical in their general shape but in which they vary somewhat in size. This finished product may be withdrawn and used advantageously without further treatment. However, for convenience in operation we prefer to separate the granular carbon black thus formed into two lots of relatively coarse and fine grains, as by sifting it through mesh of suitable size. The proportion of carbon black in these portions depends upon the size of the mesh of the sifting screen used and we have operated very satisfactorily with a mesh which results in two substantially equal portions. The coarser portion may be withdrawn at this stage as finished product, while the fine portion may be used as a priming charge for the succeeding batch. The fine portion is, accordingly, returned to the chamber 80 and sufficient untreated carbon black added to form a new batch.

While we have referred to the treatment of each batch as requiring a period of from 60 to 120 minutes, this may be varied within certain limits. It should be noted, however, that if the coarse size grains are not separated in the manner above described, then the grains formed may exceed the stable dimensions and will thereupon be broken down into fragments of more or less irregular shape which are substantially free of dust.

It will be noted that the two skeleton cages 36 and 40 are disposed in telescopic relation and that their impelling or baffle elements 32 and 42 are alternately arranged in radially spaced relation. The distance between these cooperating elements appears to be of some importance in carrying out our process and we have found that a radial spacing or clearance of about one inch between adjacent elements 32 and 42 is quite satisfactory. The spiral straps 38 and 46 are arranged in a similar manner and may be located on the outer sides of their baffle elements for convenience in manufacturing the cages. The speed of relative rotation between the cages is also of importance in successfully carrying out the process and, as above stated, we have found that a relative rotation between 27 and 45 R. P. M. is very satisfactory.

In addition to the characteristics of relatively high density and non-coherent surface, the carbon black granules produced as above outlined

are relatively tenacious in their structure. They cannot be crushed between the fingers by normal pressure, although they may be readily crushed against a hard surface or by rubbing which subjects them to shearing stress between the fingers.

While we have explained to some extent a theory of the formation of the dense spherical granules which it is the object of our invention to produce, it should be understood that the exact nature and cause of the results attained are not at present fully understood and that there are undoubtedly other important factors in the phenomenon which will become apparent upon further practice of the process herein disclosed.

It is believed, however, that never heretofore has flocculent carbon black been progressively built up in a dry state, without assistance of binders, solvents, liquids, tars or other additions, either temporarily or permanently present in such a manner as to transform flocculent carbon black into substantially dustless tenacious granules, the particles of which are united only by their own cohesive force with sufficient tenacity of structure to resist disintegration when transported in bulk.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A process of compacting dry flocculent carbon black, which consists in turbulently agitating the carbon black in a dry state in a manner causing agglomeration of the particles into relatively tenacious, self sustaining and dense grains of substantially spherical shape that can be transportable in bulk without disintegration, and then agitating the agglomerated grains thus formed until they are broken down into substantially dustless fragments of irregular shape, said fragments retaining substantially the same physical properties as the dense grains.

2. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in impacting the particles of flocculent carbon black in a dry state between opposing surfaces relatively movable toward and past each other in a manner maintaining the particles in a state of pronounced turbulence and thus causing systematic and progressive agglomeration of the particles into tenacious granules of the character described.

3. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in impacting the particles of flocculent carbon black in a dry state between opposing surfaces relatively movable toward and past each other in a manner maintaining the particles in a state of pronounced turbulence and thus causing a systematic and progressive building up of the particles into tenacious granules, and simultaneously moving the particles in a direction transversely to the relative movement of said surfaces, thereby keeping the product uniform throughout the mass.

4. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in turbulently impacting particles of flocculent carbon black in a dry state by propelling surfaces moving in a circular direction and opposing such movement of the particles by other surfaces located out of the path of movement of the first-mentioned surfaces, thereby systematically and progressively building up the particles into granules of the character described.

5. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in forcing groups of particles of flocculent carbon black to flow between baffles having a predetermined clearance, thereby maintaining the particles in a state of pronounced turbulence and thus subjecting certain particles to recurring impacts and systematically and progressively agglomerating them with others into non-adherent, tenacious granules of the character described.

6. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting particles of dry flocculent carbon black in a mass to violent turbulence in which occur successive and rapid impacting impulses causing the systematic and progressive agglomeration of the particles into tenacious, non-adherent granules, alternated with relaxing impulses causing a rearrangement of the particles in the mass.

7. A process of producing carbon black in the form of granules of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in causing particles of dry carbon black in a mass to flow in spaced concentric zones, and simultaneously subjecting the particles to recurring pressure in relatively transverse directions, thereby maintaining the particles in a state of pronounced turbulence for a time sufficient to cause the systematic and progressive agglomeration of the particles into substantially spherical granules of the character described.

8. A process of compacting flocculent carbon black, consisting in impacting the particles thereof in a dry state by and between opposing surfaces extending into and surrounding a mass of the particles being treated and relatively movable toward and past each other in a manner maintaining the particles in a state of pronounced turbulence and thus causing systematic and progressive agglomeration of the particles into relatively dense, non-adherent granules capable of withstanding handling in bulk without substantial disintegration, and simultaneously moving the particles and granules in a direction transversely to the relative movement of said surfaces, thereby keeping the granules substantially uniform in size throughout the mass.

9. A process of compacting carbon black, which consists in impacting the particles of flocculent carbon black in a dry state between opposing surfaces thereby maintaining the particles in a state of pronounced turbulence for a time sufficient to cause systematic and progressive agglomeration of the particles into relatively dense, non-adherent granules capable of withstanding handling in bulk without substantial disintegration, separating the resulting product into relatively coarse and fine portions, and using the relatively fine portions as a priming charge in succeeding operations.

10. A process of producing carbon black in the form of granules of pure carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting flocculent carbon black of commerce in a dry state to turbulent agitation, and con-

tinuing such agitation until said carbon black assumes the form of relatively tenacious self-sustaining granules.

11. A process of producing carbon black in the form of granules of pure carbon black of sufficient cohesion to withstand handling in bulk without disintegration, which consists in subjecting particles of flocculent carbon black in a dry state to multi-directional impact with other carbon black particles, by turbulent agitation of a dry mass of flocculent carbon black, and continuing such agitation until said carbon black agglomerates into granules which are self-sustaining but disintegrate readily under shearing pressure.

12. A process of producing carbon black in granular condition, which consists in mixing a charge of the flocculent carbon black of commerce with agglomerated granules of carbon black and then subjecting the said granules to multi-directional impact with particles of the flocculent carbon black by turbulent agitation of the mixture, and continuing such agitation until particles of flocculent carbon black agglomerate with the granular carbon black to form a carbon black in the form of granules capable of withstanding handling in bulk without disintegration.

13. The process of manufacturing substantially spherical grain carbon black, which consists in maintaining dry particles of flocculent carbon black in a state approaching perfect turbulence, whereby the particles are freely subjected to symmetrical impact and caused to cohere to other granules and are thus built up progressively into relatively dustless, substantially spherical granules which in bulk have a low viscosity and a density of over 20 pounds per cubic foot.

14. The process of manufacturing spherical grain carbon black which consists in turbulently agitating a dry charge of flocculent carbon black particles at moderate temperature, in such a manner as to subject certain particles symmetrically to free impact pressure of other particles, thus building them up progressively and systematically into substantially spherical granules of an order of approximately 200 mesh in diameter and capable of withstanding handling in bulk without substantial disintegration.

15. The process of imparting dense granular and tenacious characteristics to the light, fluffy carbon black of commerce, which consists in subjecting particles of the latter, in a dry state, to turbulent agitation, thus compacting the particles by impact and causing them to unite by impact-generated cohesion into granules of pure carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration.

16. The process of producing a dense granular carbon black which consists in mixing a charge of flocculent carbon black with carbon black which has been agglomerated into granules and densified, and then turbulently agitating the mixture to cause the systematic and progressive agglomeration of the flocculent carbon black upon said granules into larger, substantially spherical granules, and then applying repeated multi-directional pressure to such granules imparting a smooth surface finish thereto and making them capable of withstanding handling in bulk without substantial disintegration.

17. The process of producing dense, spherical carbon black which consists in mixing a charge of flocculent carbon black with granular carbon black and then turbulently agitating the mixture in a dry condition so that the flocculent particles contact with the granules with sufficient velocity to unite therewith to form substantially spherical granules of pure carbon black which in bulk have a low viscosity and a density of over 20 pounds per cubic foot.

18. The process of producing a dense, dustless granular carbon black which consists in mixing a charge of flocculent carbon black with a priming of granular carbon black, and then turbulently agitating the mixture at a temperature below that at which oxidation of the carbon black occurs to cause the flocculent carbon black to agglomerate with the granular carbon black and by subjecting the agglomerates to intermittent multi-directional pressure, to convert them into substantially spherical grain form sufficiently tenacious to withstand substantial normal pressure but being readily disintegrated by shearing pressure.

19. A process of producing substantially dustless, free flowing granules of pure carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in mixing a charge of the flocculent carbon black of commerce with small agglomerated granules of carbon black, and then subjecting the mixture to turbulent agitation and continuing such agitation until said mixture has taken the form of relatively tenacious, self-sustaining granules.

20. A process of producing carbon black in the form of a substantially dustless free flowing mass of agglomerates of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting particles of flocculent carbon black in a dry state to turbulent agitation by rolling and tumbling and thereby producing said substantially dustless mass of the character described.

21. A process of producing from a mass of flocculent carbon black in the dry, loose state in which it is delivered from the ordinary bolting and sifting apparatus of a carbon black plant, a substantially dustless mass of free flowing coherent agglomerates of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting such flocculent carbon black to turbulent agitation, and continuing such agitation until such carbon black assumes the form of said relatively tenacious self-sustaining granules.

22. A process of producing from a mass of flocculent carbon black in the dry, loose state in which it is delivered from the ordinary bolting and sifting apparatus of a carbon black plant, a substantially dustless mass of free flowing coherent agglomerates of commercially pure carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in adding agglomerated granules of carbon black to a mass of the flocculent carbon black and subjecting the mixture to turbulent agitation by rolling and tumbling.

23. A process of transforming a mass of dry, flocculent carbon black into a substantially dustless mass of free flowing, relatively tenacious, self-sustaining granules of substantial density and cohesion, which consists in adding agglomerated granules of carbon black to a mass of flocculent carbon black and then subjecting the resulting mixture to turbulent agitation to form therein and therefrom granules of carbon black of the character described which are capable of

withstanding handling in bulk without substantial disintegration.

24. A process of producing from a mass of flocculent carbon black in the dry, loose state in which it is delivered from the ordinary bolting and sifting apparatus of a carbon black plant, a substantially dustless mass of free flowing coherent agglomerates of carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting such flocculent carbon black to turbulent agitation, and continuing such agitation until substantially all the flocculent carbon black is transformed into relatively tenacious self-sustaining granules having an apparent density of over 20 lbs. per cubic foot.

25. A process of transforming a mass of flocculent carbon black into a substantially dustless, free flowing mass of relatively tenacious, self-sustaining granules, transportable in bulk without disintegration, which consists in subjecting said mass to turbulent agitation and thereby agglomerating substantially all of said mass of flocculent black into relatively compact granules of the character described, substantially free in bulk of unagglomerated particles and having an apparent density of not less than 20 lbs. per cubic foot.

26. The process of transforming flocculent, powdery carbon black into a collection of relatively sturdy, self-sustaining granules, said collection being characterized by being free flowing and substantially free from unagglomerated particles or dust, which consists in subjecting said flocculent carbon black to turbulent agitation whereby the particles of said carbon black are brought into multi-directional bombarding relation with each other, causing impact-generated cohesions between particles of such strength as to cause the particles to build up into tenacious granules, and continuing said turbulent agitation until said granules are sufficiently sturdy to withstand handling in bulk without disintegration.

27. The process of making granular carbon black, said product being characterized by being free flowing and substantially free of unagglomerated particles or dust, substantially as described, which consists in adding agglomerated granules of carbon black to a mass of flocculent powdery carbon black, and subjecting said mixture to turbulent agitation thereby creating multi-directional impacts between particles and granules, whereby the particles are caused by impact-generated cohesions to cohere to the said granules or to each other, and continuing said turbulent agitation until all the dusty particles have been thus caused to cohere into granules sufficiently sturdy to withstand handling in bulk without disintegration.

28. A process of producing carbon black in the form of granules of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting flocculent carbon black of commerce in a dry state to turbulent agitation by lifting the carbon black in one area after another and allowing it to fall freely in a closed container and continuing such agitation until the black assumes the form of relatively tenacious self-sustaining granules.

29. A process of producing carbon black in the form of granules of sufficient density and cohesion to withstand handling in bulk without disintegration which consists in subjecting flocculent carbon black of commerce in a dry state to turbulent agitation by passing a series of blades angularly through a mass thereof in a closed container, and continuing such agitation until the carbon black assumes the form of relatively tenacious self-sustaining granules.

EDMUND BILLINGS.
HAROLD H. OFFUTT.

CERTIFICATE OF CORRECTION.

Patent No. 2,120,541. June 14, 1938.

EDMUND BILLINGS, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, lines 52 and 53, strike out the words "under conditions of turbulent pressure such"; page 2, second column, line 63, for "primary" read priming; page 3, first column, line 66, strike out the word "stationary" and insert the same before the syllable "spi-" in line 67; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 19th day of July, A. D. 1938.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.



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Nov. 12, 1935. E. BILLINGS ET AL Re. 19,750

MANUFACTURE OF PRODUCTS OF CARBON BLACK AND SIMILAR FINE POWDERS

Original Filed Aug. 12, 1933

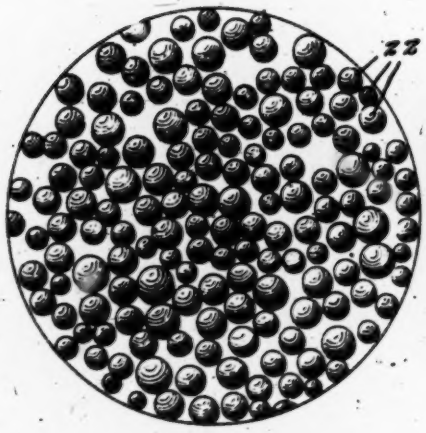


Fig.1.

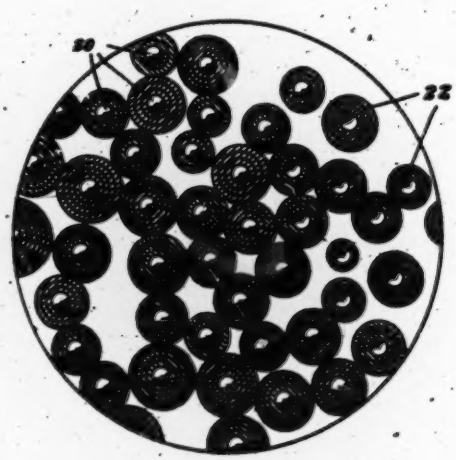


Fig.2.



Fig.3.



Fig.4.



Fig.6.



Fig.5.

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Reissued Nov. 12, 1935 Re. 19,750

UNITED STATES PATENT OFFICE

19,750

MANUFACTURE OF PRODUCTS OF CARBON BLACK AND SIMILAR FINE POWDERS

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Original No. 1,957,314, dated May 1, 1934, Serial No. 684,834, August 12, 1933. Application for reissue January 19, 1935, Serial No. 2,814

13 Claims. (Cl. 134—88)

This invention relates to a novel product made from fine dry powders as a result of processes of mechanical treatment solely. In general it applies to the broad field of solid substances which derive their applications in the arts and sciences from their fine state of sub-division. This extreme degree of fineness carries the inevitable and generally undesirable concomitant of dustiness, and this dustiness, arising from the minute weight of the individual particle, may lead to serious undesirable commercial consequences; for example, contamination of other nearby products and processes, excessive deterioration of fixtures, machinery and furnishings and respiratory and other occupational disorders in the workmen exposed to the dust.

We have discovered that certain powdered materials of this type may be agglomerated on themselves, by purely mechanical treatment, without the assistance of binders, solvents, liquids, tars, or the addition, either temporarily or permanently, of any foreign substance. The agglomerates formed by our process are of greater apparent density in bulk than the loose powders from which they are formed and consist of more or less spherical masses having such a high ratio of weight to surface that they are not dusty. Also, they have smooth, polished, non-adherent surfaces and compact, adhesive internal structures. These characteristics give the resulting agglomerates a free-flowing nature which materially simplifies handling in bulk, homogeneous mixing with other materials, delivery by gravity through chutes and pipelines, packaging in any desired weight, and all of the other conveniences imparted to the product by its extreme fluidity and dustlessness.

One important field of use for our invention is the carbon black industry, for example, in connection with the so-called "channel" carbon black produced by burning natural gas flames against metallic surfaces. The ultimate carbon black particle is so small that its shape and exact size are beyond microscopic determination. As first produced, such carbon black has an apparent density in bulk of about three pounds per cubic foot. It has been the practice to increase this apparent density in bulk to about twelve pounds per cubic foot by stirring and then to increase it further to about twenty-five pounds per cubic foot by compressing. The resulting compressed carbon black due to its dusty nature is still inconvenient to handle because it so readily reverts to its dusty form. In addition to this, the difficulties of packaging carbon black by compression have necessitated the employment of small sacks, twenty-five pounds or less in capacity, and for practical reasons the industry has obliged to standardize on two sizes of sacks.

We have found that by subjecting the carbon black of commerce to certain conditions of turbulent pressure, the fine dusty particles of which it is composed may be caused to adhere and agglomerate firmly with each other in groups. Thus the carbon black may be converted into small spherical granules of apparent density in bulk higher than before such treatment, of relatively tenacious structure, with each of such granules presenting a more or less polished, non-adherent surface. Carbon black in this spherical grain form possesses all of the advantages of convenience in handling and distribution above discussed and, in addition, it disperses perfectly in rubber mixtures, and is otherwise as useful or more useful in rubber making and other industries than the compressed carbon black of commerce formerly employed.

Similarly we have found that a number of other fine, dry powders, among them, all of the carbon pigments commonly known as lamp blacks, zinc oxide, iron oxide and clay, (previously reduced to the requisite state of sub-division if necessary) have the property of agglomerating under determinable conditions of mechanical manipulation and that when subjected to suitable turbulent pressure they may be converted into small, tenacious spherical granules of concentric shelled structure and having non-adherent surfaces.

The powders which display this behaviour have certain other characteristics which serve to differentiate them from those powders which cannot be agglomerated by such treatment. For example, if jigged on a vibrating surface, they will agglomerate into small spheres of very fragile structure, they will form an adhesive lump when squeezed, they will adhere to metallic or non-metallic rods when these are forcibly drawn through them and they are free from gummy and resinous ingredients. The present invention has to do with fine powders or pigments having these properties or characteristics and for convenience they will be termed herein as "agglomerative powders"

We have discovered that by subjecting them to the proper conditions of turbulent pressure, and to proper manipulation these agglomerative powders may be converted into spherical granules of greatly increased apparent density, having also internal structures of spherical shells concentrically disposed about central cores, with each granule presenting a smooth, polished, non-ad-

herent and non-coherent exterior surface. The self-generated spherical granules thus produced are of appreciable mechanical strength and, since they have the property of withstanding very considerable direct pressure, they may be handled or transported in bulk with practically no disintegration. The powder, converted into this spherical-grain form, is substantially dustless, free-flowing and non-adherent.

The proper conditions of turbulent pressure necessary for the conversion of agglomerative powders into such spherical granules may be created by the use of various types of mechanical apparatus. One example of suitable apparatus is disclosed in our pending application Serial No. 642,280, filed Nov. 16, 1932. We have found that, under certain conditions, we can directly convert fine agglomerative powders from their loose, seemingly amorphous form into this dense, spherical-grain form. However, the process is usually more rapid, economical and commercially efficient if a priming charge of pre-formed spherical material is mixed with the powder being treated. This priming material may be either the same or different from the powder. If such a priming charge is not used, the first effect of turbulent pressure on the powder may be to cause the gradual formation of a small number of nuclei consisting of a number of powder particles adherently bonded together. Since, in an adequate space of time under turbulent pressure, the nuclei as they form are pressed upon from all sides and in all directions, their growth is in three equal dimensions, thus their form is spherical.

The priming charge serves at least two important purposes, i. e. (1) It immediately provides cores or nuclei upon which the powder may at once begin to adhere thus promoting or expediting formation of a large proportion of spherical, well-agglomerated granules of low viscosity. The resulting lower average viscosity enhances the turbulent effect of the mechanical apparatus used and thereby accelerates the conversion from powder to dustless spherical agglomerate. (2) It provides immediately bodies of much larger weight than the weight of the particles of dust. These larger bodies, by collision, provide impacts on the dust particles of sufficient intensity to cause permanent adhesion. They also widen the zones of effective action in the apparatus and so permit the use of greater clearances than would otherwise be the case, thereby simplifying the mechanical construction of the apparatus.

There are a number of ways of producing satisfactory priming charge material but all are alternative methods for accomplishing two purposes; first, exerting sufficient pressure on the powder particles to form firm adhesions; second reduction of the agglomerates thus formed to a convenient and suitable size and with three appreciable dimensions. For example, satisfactory cores may be secured from some agglomerative powders by subjecting relatively thin layers of the powders to direct compression, sufficiently heavy to cause the formation of a chunk, cake or briquette. This, if then rubbed through a wire screen of suitable mesh, is partially disintegrated and the resulting product of this treatment is suitable for core material and may be readily built up into spherical form by the addition of concentric external layers or shells.

As another example, spheres of foreign material of suitable density and size, may be mixed with the agglomerative powder and the mixture subjected to turbulent pressure. The powder will then form a substantially concentric, spherical shell on each sphere. If the composite spherical granules thus formed are rubbed upon a screen of the proper mesh to exclude the original spheres, they will break and fragments of the shells passing through the screen will be found sufficiently dense and adhesive to serve as priming material for the subsequent agglomeration of powders of their own nature. Examples of spheres suitable for this purpose are fine shot or seeds, such as those of the beet, clover or petunia.

Likewise composite granules may be formed by using cores made from one powder with shells formed from an entirely different powder, for use where a free-flowing dustless mixture of two or more such powders is commercially desired.

An important feature of our invention lies in our discovery of the fact that we are able to take those small agglomerates, which may be so fine as to partake of the characteristics of dust, and build them, through successive addition of shells, to a size that imparts to the product the commercial advantages above discussed, viz., dustlessness, high density, low mass viscosity, spherical shape and non-adherent surface.

Another important feature of our invention lies in our discovery of the further fact that we can alter and control the degree of surface polish imparted to the granules since, under proper conditions, this is a function of time of treatment.

Our ability to control the degree of surface polish enables us:

1—To control the viscosity of the material in bulk, thus facilitating handling.

2—To offset the increase in viscosity in bulk which would otherwise naturally accompany increase in average diameter of the individual spheres.

Thus, ability to control polish enables us to maintain adequate turbulent pressure throughout a wide range of average sphere diameter and thus to add a greater number of concentric shells and produce larger and more useful spherical agglomerates.

Since the impacts delivered by one sphere to another are a function of the weight of the spheres, it becomes possible to increase rapidly the density of the spherical granules in process of treatment by employing a priming charge of large spheres. We may, by proper manipulation and adequate time of treatment, thus cause the density of granules to approach the absolute specific gravity of the material being treated. This is another characteristic and extremely important feature of our invention.

For purposes of illustration, the invention will now be more specifically described as carried out in connection with the production of spherical grain carbon black. In the accompanying drawing,

Fig. 1 represents spherical grain carbon black as seen in the field of a microscope, magnified 20 diameters.

Fig. 2 represents spherical grain zinc oxide, the granules being shown in cross section in the field of a microscope, magnified 60 diameters.

Figs. 3 and 4 are sectional views of single spherical granules, magnified 200 diameters; and

Figs. 5 and 6 represent diagrammatically the

conversion process as carried out by one form of suitable apparatus.

In the preparation of spherical grain carbon black from the carbon black of commerce, the cores may be first produced separately by subjecting the seemingly amorphous powder to prolonged treatment under conditions of adequate turbulent pressure. The cores thus produced present a smooth, non-adherent surface and may conveniently have an apparent density in bulk of more than 12 pounds per cubic foot, for example, about 18 to 20 pounds per cubic foot. The average size of these cores, as measured by sieve tests, is determined by the degrees of turbulence and pressure to which the powder is subjected. The optimum size is determined by the number of concentric shell-like layers which are subsequently to be added to the core and by the design of the apparatus in which the additions are made. For carbon black, an average core diameter ranging from 300 mesh to 60 mesh has been found satisfactory.

Substantially equal amounts of these cores and of commercial carbon black may now be introduced into an apparatus such, for example, as that disclosed in our pending application Serial No. 642,850. This apparatus comprises a drum which constitutes a container and in which are arranged a plurality of impelling elements and baffle elements movable past each other in an approaching and receding manner and provided with means to cause also an up-and-down circulation in the mass delivered thereto. The composite charge may be thus subjected to pressure and to turbulence causing this pressure to be multi-directional upon each particle thereof. This is accompanied by a sliding or shifting action of the mass upon itself under conditions of pressure and a stroking or polishing interaction of its particles. Under these conditions there occurs an agglomeration of the flocculent carbon black upon the cores or fines with the result that a spherical shell is built up upon each core.

In Fig. 4 of the drawing is shown a core 10 upon which has been formed a concentric shell 11. This figure represents a single granule of spherical-grain carbon black at high magnification, the core being shown in elevation and the concentric shells in section.

The process may be continued until all of the flocculent carbon black has been agglomerated upon the cores in the form of spherical shells and if the components of the initial charges are substantially equal, it is apparent that each shell will comprise substantially 50% of the weight of the spherical grain thus formed. It is desirable to continue the process for a time after the disappearance of the flocculent carbon black from the apparatus sufficient to polish the surfaces of the spherical grains. At this stage the entire charge has been converted to spherical granules of high apparent density, the individual granules being sufficiently tenacious to stand such pressures as they may be subjected to in handling, and presenting a more or less polished non-adherent surface. If the process is continued further, the granules tend to become more and more dense and to be reduced in diameter. The process may be stopped when the spherical granules have thus reached a convenient commercial density and polish.

We have described the production of spherical granules comprising a core and a single concentric shell by a single regenerative process terminating when an equal weight of flocculent carbon black has been agglomerated upon each core. If desired, a new charge of flocculent carbon black may now be introduced into the apparatus and, as before, this may approximately equal in weight the amount of the spherical grain charge already produced or remaining therein. The process is now renewed as before and a second concentric shell agglomerated upon the outer surface of the first. Such a second shell is indicated by reference character 12 in Fig. 4 of the drawing. When the flocculent carbon black of the charge has again agglomerated, the process may be repeated. In Fig. 4 a third concentric shell 13 is shown and in Fig. 2 are shown granules having as many as six concentric shells agglomerated about the core. What has already been said applies to these additional regenerative processes. The core and each shell is defined by a polished or somewhat glazed spherical surface upon which the next larger shell is agglomerated and to which it directly coheres. These surfaces may be readily detected as surfaces of cleavage or division in partially crushed or sectioned spherical granules.

Fig. 1 represents spherical-grain carbon black or of other agglomerative powders as seen in the field of a microscope in the magnification of 50 diameters. It will be noted that all the grains are spherical or substantially so and that there is a substantially uniform upper limit of diameter which has been attained by a large number of the granules. The smaller granules represent those which contain in their composition a smaller number of concentric shells than the larger granules, or the shells of which are thinner.

We have found that in the case of carbon black subjected to treatment in the type of apparatus described in our pending application Serial No. 642,850, it is best to use a priming charge not coarser than 60 mesh if an increment of equal volume is to be added in the form of a shell, because the resulting spherical grains produced by such an increment will then have a size of about 40 mesh and, using carbon black, we have found that the subjection to turbulent pressure of a charge of spherical grain carbon black containing a substantial percentage of grains substantially larger than 40 mesh in the type of apparatus described, results in viscosities and pressures so great as to crack and burst the granular agglomerates, thus reproducing sticky, dusty material from the free-flowing, dustless charge. For this reason, when treating carbon black by the above process, we have found it advantageous to start with a priming charge in the range of 150 mesh so that a large number of concentric shells could be added before reaching the limiting size.

As the diameter of the spherical grains increases with the addition to them of successive increments of carbon black, and also as the application of turbulent pressure is continued after all the flocculent carbon black has been applied to the cores of the priming charge, some of the shells become ruptured into fragments but not into dust, and these fragments, in the presence of their neighbors, round off and become to all intents and purposes new core material. This process continues throughout the run and results in a decrease of the average size of the agglomerates with time of running. It is also to be understood that it is the natural tendency of all these agglomerates to become smaller with time of running because of increase in density due to the impacts and pressures applied to their surfaces.

At the conclusion of the first cycle of the operation the finished charge may be withdrawn from the apparatus and in this condition constitutes a satisfactory and valuable commercial product. It is usually preferable, however, to lift the material at the end of each cycle, separating the smaller agglomerates from the mass so that they may be used as priming material for new charges, and retaining in the finished product only agglomerates of the larger sizes.

In the light of our present knowledge, we may suggest one theory of what takes place within the drum to cause the carbon black to assume its new and relatively dense form by reference to Figs. 5 and 6 of the drawing. The stationary elements 42 are arranged alternately with the moving elements 32 and the charge of black surrounds and entirely fills the space between these elements. As the elements 32 move relative to the elements 42 (Fig. 5), they tend to carry the black in a mass therewith but such movement of the black is opposed by the stationary elements 42. The result is that cones 86 and 87 of black form both forwardly and rearwardly of the elements 32 and move along with these elements, while cones 88 and 89 of black form both forwardly and rearwardly of the elements 42 remain stationary therewith. The carbon black intermediate the elements 32 and 42 (indicated by line 90 of black particles in Fig. 5) is in a state of turbulence, the black adjacent to the elements 32 moving nearly as fast as those elements and the carbon black adjacent to the elements 42 remaining nearly stationary. The relative positions of the elements are constantly changing as the elements 32 approach and recede from the elements 42 and, due to this action, the particles of carbon black are alternatively being brought into most intimate and bombarding relation whereby they are compacted into the relatively dense form of the finished product and into a relatively loose relation, wherein they are free to rearrange themselves for the next impact. Thus the black particles are intermittently subjected to pressures or impacts and are relatively rearranged between the successive impacts, such impacts or pressures thereby becoming multi-directional, i. e. not occurring twice in succession in the same direction.

If the cores are formed from compressed or briquetted material we have found it advantageous to subject the rough fragments resulting from this method to turbulent pressure for a short period. This treatment breaks off the rough irregular projections of such cores, tends to equalize the three dimensions, fills in the cavities with the detritus resulting from the abrasion of the projections and thus produces cores which are more nearly perfect spheres. This condition is desirable because the more nearly spherical the priming charge, the lower the viscosity of the subsequent mixture with the agglomerative powder. Lower viscosity, within limits, enhances the turbulent pressure effects of our apparatus and thus assists the conversion process. If the core is spherical to start with, the shell agglomerated thereon will also be spherical. If the core is irregular in its shape, the shell will not be perfectly spherical but will tend to approach spherical shape more nearly than the core. In Fig. 3 is shown a core 14 of more or less irregular shape, such as might be found by the compressing and breaking process, and upon this has been formed a shell 15. This, it will be noted tends to fill in and smooth out the surface irregularities of the core and to approach spherical shape.

Fig. 2 represents spherical granules of zinc oxide which have been imbedded in a transparent medium and then sectioned, as seen in the field of a microscope in the magnification of 60 diameters. In this view, the cores 20 of certain granules are shown in place where as other granules 22 are shown as having lost their cores. These were displaced and lost in the operation of sectioning the spherical granules.

The limiting size of the agglomerates depends somewhat upon the design and operation of the agglomerating apparatus used for producing the requisite turbulent pressures, but is even more dependent upon the nature of the agglomerative powder being treated. Zinc oxide, for example, can be caused to agglomerate by increments into spheres of larger than 30 mesh before the resulting increased pressure exceeds the structural resistance of the spherical grains and break-down into dust ensues.

The continuous tendency toward the replacement of fine material as the process is carried out is advantageous for two reasons:

1. It maintains the distribution of particle sizes.

2. It affords a continuous replacement of fine material for the start of new cycles if these fine particles are screened out at the end of each run, thus removing the necessity for a separate and distinct process or step for the production of cores.

The distribution of particle sizes is important to the process for three reasons:

1. It decreases the viscosity of the charge by providing supporting surfaces in the interstices between the larger particles, thus increasing the ability of the apparatus to produce adequate turbulent pressures.

2. It decreases the liability of the material to be ruptured or crushed by conveying, transportation, or handling, thus inhibiting the premature return of the spherical grains to their original, dusty fine components.

3. It increases the apparent density in bulk of the commercial product over that which would obtain if only one size of particle were present, thus economizing on packing materials and storage space per unit weight.

While in describing the process of our invention as carried out in connection with carbon black, we have suggested the addition of an equal weight of flocculent material to the priming charge, it is entirely practicable to double this proportion, that is, to add flocculent carbon black in an amount equal to twice the weight of the priming charge. The percentage of priming material necessary or advisable in the charge is a function of the combined viscosities of the priming material and the fine powder. From this it follows that it is possible to use a 5% or 10% priming charge in treating certain powders, whereas a 33% priming charge may be required in treating other fine powders. In the case of carbon black, a 33% priming charge is the minimum below which it is not convenient to go.

This application is, in part, a continuation of our earlier application, Serial No. 623,184, filed July 18, 1932, and corresponding to our Canadian patent, No. 333,741, granted July 4, 1933.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent of the United States is:

1. As a new product of manufacture, carbon

black in the form of spherical granules having a relatively tenacious internal structure consisting of concentric shells of progressively increasing diameters bonded together with sufficient tenacity to withstand transportation in bulk without distintegration, the granules presenting polished exterior surfaces and having an apparent density in bulk of more than 12 pounds per cubic foot.

2. As a new product of manufacture, carbon black in the form of spherical granules comprising in internal structure a central core with a plurality of concentric spherical shells enclosing the same and united to each other with sufficient tenacity to withstand disintegration when transported in bulk, the granules presenting smooth, non-adherent exterior surfaces and having an apparent density in bulk of at least 18 pounds per cubic foot.

3. As a manufactured product, carbon black in the form of spherical granules between 300 mesh and 20 mesh in diameter, of more than 12 pounds per cubic foot in apparent density and each comprising a dense compact core surrounded by a spherical shell of substantially the same density as the core and built up progressively upon the core by the application of turbulent pressure to flocculent carbon black with sufficient cohesiveness to maintain its identity and adapt the granules to withstand disintegration when transported in bulk and presenting a smooth, non-adherent exterior surface.

4. As a manufactured product, carbon black in the form of spherical granules each comprising an integral coherent core surrounded by a series of concentric shells of substantially equal density, each sufficiently cohesive to maintain its identity when the granule is cut in cross section, presenting a spherical exterior light-reflecting surface and being tenaciously bonded with the core sufficiently to withstand disintegration when transported in bulk.

5. A new product of manufacture, consisting of carbon black in the form of solid spherical granules between 300 mesh and 20 mesh in diameter, having hard, impact-formed, non-adherent surfaces and a compact, dense structure of pure carbon black particles built up systematically and progressively by application of turbulent pressure to flocculent carbon black, the particles being united only by their own cohesive force with sufficient tenacity of structure to resist disintegration when transported in bulk, and having a density of more than 12 pounds per cubic foot.

6. A new product of manufacture, consisting of carbon black in the form of solid spherical granules between 300 mesh and 20 mesh in diameter and each having a smooth, impact-formed, non-adherent surface and a dense sturdy structure built up progressively and systematically by application of turbulent pressure to flocculent carbon black, and having an apparent density of more than 12 pounds per cubic foot.

7. A new product of manufacture, consisting of carbon black in the form of substantially spherical granules between 300 mesh and 20 mesh in diameter, and fragments of such spherical granules, said spherical granules having polished non-adherent surfaces, an apparent density of not less than 12 pounds per cubic foot and a compact dense internal structure of pure carbon black particles arranged systematically in forcible engagement with each other by turbulent pressure and maintained by their own cohesive force with such tenacity of structure as to withstand disintegration when transported in bulk.

8. As a new product of manufacture, fine dry agglomerative powder in the form of solid, tenacious, substantially spherical granules not over 20-mesh in diameter and comprising in their internal structure distinct concentric shells of progressively increasing diameter bonded together only by the impact-generated cohesion of the particles of the powder, the granules presenting polished exterior surfaces and being adapted by reason of their structure to withstand transportation in bulk without disintegration.

9. As a new product of manufacture, fine dry agglomerative powder in the form of solid, substantially spherical granules comprising in their internal structure concentric shells, each intact, of substantially equal density and bonded together only by the impact-generated cohesion of the particles of the powder with sufficient tenacity to withstand the pressure of transportation in bulk without substantial disintegration and presenting a polished, non-adherent exterior surface.

10. As a new product of manufacture, fine dry agglomerative powder in the form of solid, substantially spherical granules of sufficient mechanical strength to withstand disintegration when handled in bulk and of greater apparent density than the loose powder, each granule comprising a series of distinct impact-formed concentric shells united in an integral body only by the impact-generated cohesion of the particles of the powder and presenting a smooth non-adherent surface.

11. A new product of manufacture, consisting of fine dry agglomerative powder in the form of solid, substantially spherical granules between 300-mesh and 20-mesh in diameter, having hard impact-formed non-adherent surfaces and a compact dense structure of the pure agglomerative powder built up systematically and progressively by the application of turbulent pressure to the dry powder, the particles being united only by their own cohesive force with sufficient tenacity of structure to resist disintegration when transported in bulk, and having an apparent density in bulk greater than that of the powder.

12. As a manufactured product, a fine dry powder of zinc oxide agglomerated in the form of spherical granules having an internal structure sufficiently tenacious to withstand disintegration when handled in bulk and consisting of a substantially spherical core and a concentric shell built up progressively and systematically by the application of turbulent pressure to the zinc oxide powder and united thereto by impact-generated cohesion of the zinc oxide particles and presenting a polished exterior surface.

13. As a manufactured product, a fine dry powder of zinc oxide agglomerated in the form of solid spherical granules between 300-mesh and 20-mesh in diameter and each having a smooth impact-formed non-adherent surface and a dense sturdy structure built up progressively and systematically by the application of turbulent pressure to the free dry powder of zinc oxide and having an apparent density of more than 20 pounds per cubic foot.

EDMUND BILLINGS.
HAROLD H. OFFUTT.

Nov. 1, 1938. H. H. OFFUTT 2,134,950

GRAPHITIZED CARBON BLACK

Original Filed Aug. 20, 1934



Fig. 1.



Fig. 3.



Fig. 2.

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Patented Nov. 1, 1938

2,134,950

UNITED STATES PATENT OFFICE

2,134,950

GRAPHITIZED CARBON BLACK

Harold H. Offutt, Winchester, Mass., assignor to Godfrey L. Cabot, Inc., Boston, Mass., a corporation of Massachusetts

Application August 20, 1934, Serial No. 740,681
Renewed November 3, 1936

15 Claims. (Cl. 23—209)

This invention relates to a novel material or product of manufacture comprising graphitized carbon black in the form of relatively dense, free-flowing granules. It includes within its scope a novel process of producing graphitized carbon black in this form from the flocculent carbon black of commerce.

One important field of use of my invention is in connection with flocculent carbon black made by the so-called channel or impingement process, in which natural gas is burned against metallic surfaces. As first produced, such carbon black has an apparent density in bulk of about four pounds per cubic foot. It has been the practice to increase this apparent density in bulk to about twelve pounds per cubic foot by stirring, and then to increase it further by compressing in packages. The resulting compressed carbon black of commerce, on account of its dusty nature and bulk is still inconvenient to handle and process. Attempts have been made to graphitize such carbon black by subjecting it to intense heat in crucibles but heretofore the dust nuisance and bulky effect of handling carbon black in this form has rendered it impractical from a commercial standpoint. Indeed the difficulties of graphitizing commercial compressed carbon black have been so great that organizations having the crucible equipment and necessary electrical energy available have declined to attempt carrying out the process.

I have discovered that carbon black may be successfully graphitized and all the difficulties of handling flocculent carbon black successfully avoided by first converting the carbon black to granular form, that is, into the form of hard, dense, dustless aggregates, and then converting this granular material, a step product, to a graphitized form by heating the same in crucibles. One possible theory for accounting for this hitherto unknown property of granular carbon black, viz., its adaptability to graphitizing, is that in this form it may release its entrained air or gases more readily and completely than does carbon black in flocculent form and that the graphitizing operation is facilitated or expedited by such separation.

I prefer to convert compressed or flocculent carbon black into the form commercially known as "Spheron", that is, into substantially spherical granules, or fragments thereof, of the type disclosed in U. S. Patent No. 1,957,314, granted May 1, 1934 on an application of Billings and Offutt. Carbon black in this form presents the advantage of free flow, relatively high density, dustlessness and convenience in handling to such an extent that it may be transported in tank cars and caused to flow through pipes. It may thus be conveniently delivered to and withdrawn from the crucibles in which it is subjected to the graphitizing process. While I prefer to utilize the material, "Spheron" as a step product in carrying out my invention, I do not wish to limit myself in this respect but contemplate the graphitizing of granular carbon black of suitable characteristics however produced.

The product of my invention, viz., graphitized carbon black in granular form, has valuable properties in various uses. For example, it forms a valuable ingredient of the cathodes of dry batteries. This is usually a carbon-manganese dioxide mixture in which the carbon serves as a conductor and the manganese dioxide as a depolarizer. In such batteries, the carbon rod may be considered as a collector of current from the carbon-manganese dioxide mixture. Granulated carbon, carbon black, graphite and impalpable graphite have all been used at times in the manufacture of dry batteries. It has been found that graphitized carbon black in granular form, and particularly graphitized "Spheron", commercially known as "Graphon", is an ingredient of particularly and unexpectedly great value in the construction of dry batteries for which graphitized flocculent carbon black is not convenient to use.

In the accompanying drawing,—

Fig. 1 is a chart whereon are depicted the efficiency curves of dry batteries employing graphitized carbon black and other cathode materials;

Fig. 2 is a view, greatly magnified, showing a carbon black granule of spherical shell structure, partly in section and partly in elevation; and

Fig. 3 is a sectional view of a homogeneous granule of carbon black.

In preparing "Graphon" or graphitized "Spheron" for use in dry batteries or otherwise, the flocculent carbon black of commerce may be treated in accordance with the disclosure of U. S. Patent No. 1,957,314, above referred to. That is to say, the flocculent carbon black, compressed or otherwise, is subjected to turbulent agitation in a dry state, preferably in the presence of a priming charge of fine granular material. The impact-generated cohesion of the carbon black particles causes agglomeration of the flocculent carbon black in dense, dustless, free flowing, non-adherent granules, presenting more or less surface polish and of sufficient tenacity of structure to withstand handling in bulk without disintegration. When a priming charge is used, the resulting granules are of concentric shell structure, as

shown in Fig. 2. It is possible, however, to produce homogeneous granules of the character shown in Fig. 3, though usually less convenient to do so under commercial conditions.

The "Spheron" thus produced, comprising spherical granules from 300-mesh to 20-mesh in diameter and having a density of more than 12 pounds per cubic foot, for example from 18 to 30 pounds per cubic foot, is then placed in a crucible of suitable size and shape,—for example, one 16 feet long, having a depth of 4 feet and a width of 4 feet. The crucible and its contents are then heated gradually by electric energy to a temperature of about 2600° C., substantially out of contact with air, maintained at such temperature for a period of perhaps a week, and then gradually cooled. The time required to bring such a charge up to the requisite temperature may be perhaps six or seven days, and the time required for cooling may be the same interval, so that the graphitising step of my improved process may consume as much as three weeks for its completion. At the conclusion of this step, it will be found that the granular carbon black, which beforehand showed only traces of crystalline structure, has now been more or less completely converted to a graphitic state, although there has otherwise been no substantial change in the shape of the granules or in their apparent density.

That there has been a change in the nature or the physical structure of the carbon black is apparent from the behavior of the graphitised material, or "Graphon", in a dry battery as compared to the behavior of the ungraphitised "Spheron", as shown in the accompanying chart. Moreover, the novel product of my invention, when subjected to an X-ray test, displays a very distinct six-ring structure, whereas a similar test of the ungraphitised material exhibits a less distinct six-ring structure. That the carbon black has been at least partially converted to a graphitic form may also be demonstrated by chemical test. When "Graphon" produced as above outlined is treated with fuming nitric acid and potassium chlorate on a water bath, a pale yellow solution is obtained which, on filtering and evaporating, crystallises into colorless crystals the majority of which visibly differ from those obtained when ungraphitised carbon black is so treated.

I have specifically mentioned "Spheron" produced by mechanical manipulation in a dry state as the step product which I prefer to employ in manufacturing the novel graphitised carbon black product of my invention. I am well aware, however, that granules of carbon black may be produced from the flocculent material by various processes involving the use of one or more liquids. Granules so produced are satisfactory material for the purposes of my invention and are included within its scope.

As already intimated, an important field of use of the graphitised granular carbon black of my invention is in connection with the manufacture of dry batteries. The efficiency curves of Fig. 1 represent voltage and duration of life of three batteries, otherwise identical, but having equal amounts of "Spheron", graphite, and "Graphon" included in their cathode material.

Referring to this chart, the ordinates of the various curves represent the voltage of the battery while the abscissa represents the life of the battery in terms of minutes. It will be noted that the battery made with "Spheron" gives a potential of 1.60 volts but that its potential drops in five minutes to a 0.75 volt end point. The battery made with graphite gives an initial potential of 1.60 volts, maintains its potential for a long period, and drops off at a rather slow rate to an end point of 0.75 volt in 600 minutes. On the other hand, the battery made with "Graphon" gives the same initial potential which is maintained for a substantially longer period, and then drops off gradually to an end point of 0.75 volt at an interval very much longer than the life of any of the other batteries, viz., in about 725 minutes, indicating an increase in life of more than twenty percent over the graphite battery. These curves, therefore, indicate comparatively the greater value of my new product in the dry battery field and its value in other fields will be apparent to those skilled in the art.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. As a new product of manufacture, graphitised carbon black in the form of spherical granules having a relatively tenacious internal structure consisting of concentric shells of progressively increasing diameters bonded together with sufficient tenacity to withstand transportation in bulk without disintegration, the granules presenting polished exterior surfaces and having an apparent density in bulk of more than 12 pounds per cubic foot.

2. As a new product of manufacture, graphitised carbon black in the form of spherical granules comprising in internal structure a central core with a plurality of concentric spherical shells enclosing the same and united to each other with sufficient tenacity to withstand disintegration when transported in bulk, the granules presenting smooth, non-adherent exterior surfaces and having an apparent density in bulk of at least 18 pounds per cubic foot.

3. As a manufactured product, graphitised carbon black in the form of spherical granules between 300 mesh and 20 mesh in diameter, of more than 12 pounds per cubic foot in apparent density and each comprising a dense compact core surrounded by a spherical shell of substantially the same density as the core and built up progressively upon the core by the application of turbulent pressure to flocculent carbon black with sufficient cohesiveness to maintain its identity and adapt the granules to withstand disintegration when transported in bulk and presenting a smooth, non-adherent exterior surface.

4. As a manufactured product, graphitised carbon black in the form of spherical granules each comprising an integral coherent core surrounded by a series of concentric shells of substantially equal density, each sufficiently cohesive to maintain its identity when the granule is cut in cross section, presenting a spherical exterior light-reflecting surface and being tenaciously bonded with the core sufficiently to withstand disintegration when transported in bulk.

5. A new product of manufacture, consisting of graphitised carbon black in the form of solid spherical granules between 300 mesh and 20 mesh in diameter, having hard, impact-formed, non-adherent surfaces and a compact, dense structure of pure carbon black particles built up systematically and progressively by application of turbulent pressure to flocculent carbon black, the particles being united only by their own cohesive force with sufficient tenacity of structure to resist disintegration when transported in bulk, and having

an apparent density of more than 12 pounds per cubic foot.

6. A new product of manufacture, consisting of graphitized carbon black in the form of solid spherical granules between 300 mesh and 20 mesh in diameter and each having a smooth, impact-formed, non-adherent surface and a dense sturdy structure built up progressively and systematically by application of turbulent pressure to flocculent carbon black, and having an apparent density of more than 12 pounds per cubic foot.

7. A new product of manufacture, consisting of graphitized carbon black in the form of substantially spherical granules between 300 mesh and 20 mesh in diameter, and fragments of such spherical granules, said spherical granules having polished non-adherent surfaces, an apparent density of not less than 12 pounds per cubic foot and a compact dense internal structure of pure carbon black particles arranged systematically in forcible engagement with each other by turbulent pressure and maintained by their own cohesive force with such tenacity of structure as to withstand disintegration when transported in bulk.

8. The process of producing graphitized carbon black in the form of granules of sufficient density and cohesion to withstand handling in bulk without disintegration, which consists in subjecting flocculent carbon black of commerce in a dry state to turbulent agitation, continuing such agitation until said carbon black assumes the form of relatively tenacious, self-sustaining granules, and then subjecting said granules to a temperature of about 2000° C. for an interval of time sufficient to graphitize the material of the aggregates, meanwhile maintaining substantially unchanged the size and shape thereof.

9. The process of producing graphitized carbon black in substantially spherical grain form, which consists in maintaining dry particles of flocculent carbon black in a state approaching perfect turbulence, whereby the particles are freely subjected to symmetrical impact and caused to cohere to other granules and are thus built up progressively into relatively dustless, substantially spherical granules which in bulk have a low viscosity and an apparent density of over 20 pounds per cubic foot, and then subjecting such granules in mass to a temperature of about 2000° C. until the granules become graphitized.

10. The process of producing graphitized carbon black, which consists in first imparting dense, granular and tenacious characteristics to the light, fluffy carbon black of commerce by subjecting particles of the latter, in a dry state, to turbulent agitation, thus compacting the particles by impact and causing them to unite by impact-generated cohesion into granules of pure carbon black of sufficient density and cohesion to withstand handling in bulk without disintegration, and then heating the material so produced to graphitize it, while maintaining the size and shape of the granules substantially unchanged.

11. The process of making graphitized carbon black, which consists in converting dry flocculent carbon black into compact, substantially dustless aggregates of greater apparent density in bulk than flocculent carbon black and then subjecting the relatively dense aggregates in mass to a temperature of about 2000° C. for an interval of time sufficient to graphitize the material of the aggregates, meanwhile maintaining substantially unchanged the size or shape of the aggregates.

12. The process of making graphitized carbon black, which consists in first converting dry flocculent carbon black into dense, dustless granules, substantially spherical in shape and presenting non-adherent surfaces, and then subjecting such granules in mass to a temperature of about 2000° C. until the granules become graphitized.

13. The process of making graphitized carbon black, which consists in first subjecting dry flocculent carbon black to turbulent agitation in a dry state, causing the particles thereof to build up progressively and systematically by impact-generated cohesion into substantially spherical, dense granules and fragments thereof, and then heating the material so produced to graphitize it while maintaining the size and shape of the granules or fragments substantially unchanged.

14. The process of making graphitized carbon black, which consists in converting dry flocculent carbon black into compact, substantially dustless, granular aggregates of greater apparent density in bulk than the flocculent carbon black, and then heating said aggregates to graphitize them while maintaining the size and shape of the granular aggregate substantially unchanged.

15. A new product of manufacture, consisting of graphitized carbon black in the form of solid discrete granules between 300 mesh and 20 mesh in size and each having a smooth, non-adherent surface and sufficient density of structure to resist disintegration when transported in bulk and having an apparent density of more than twelve pounds per cubic foot.

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May 5, 1936. E. BILLINGS ET AL 2,039,766

GRANULES Or AGGLOMERATIVE POWDERS

Original Filed Aug. 12, 1933

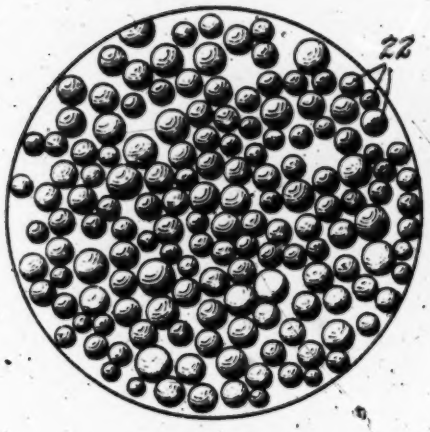


Fig. 1.

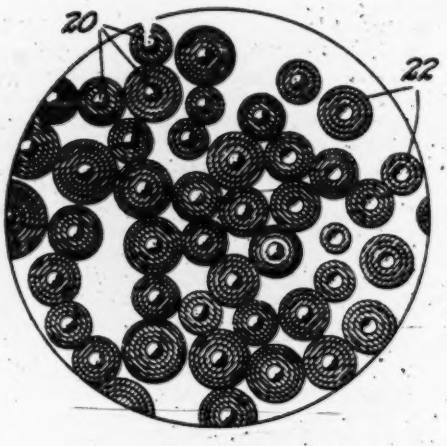


Fig. 2.



Fig. 3.



Fig. 4.

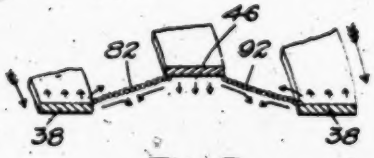


Fig. 6.

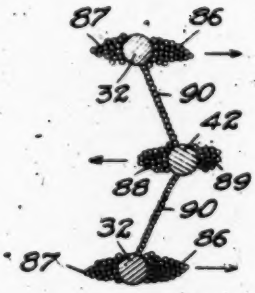


Fig. 5.

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Patented May 5, 1936

2,039,766

UNITED STATES PATENT OFFICE

2,039,766

GRANULES OF AGGLOMERATIVE POWDERS

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Original application August 12, 1933, Serial No. 684,884. Divided and this application April 19, 1934, Serial No. 721,382

2 Claims. (Cl. 134—58)

This invention relates to a novel product made from fine dry powders as a result of processes of mechanical treatment solely. In general it applies to the broad field of solid substances which derive their applications in the arts and sciences from their fine state of sub-division. This extreme degree of fineness carries the inevitable and generally undesirable concomitant of dustiness, and this dustiness, arising from the minute weight of the individual particle, may lead to serious undesirable commercial consequences; for example, contamination of other nearby products and processes, excessive deterioration of fixtures, machinery and furnishings and respiratory and other occupational disorders in the workmen exposed to the dust.

We have discovered that certain powdered materials of this type may be agglomerated on themselves, by purely mechanical treatment, without the assistance of binders, solvents, liquids, tars, or the addition, either temporarily or permanently, of any foreign substance. The agglomerates formed by our process are of greater apparent density in bulk than the loose powders from which they are formed and consist of more or less spherical masses having such a high ratio of weight to surface that they are not dusty. Also, they have smooth, polished, non-adherent surfaces and compact, adhesive internal structures. These characteristics give the resulting agglomerates a free-flowing nature which materially simplifies handling in bulk, homogeneous mixing with other materials, delivery by gravity through chutes and pipelines, packaging in any desired weight, and all of the other conveniences imparted to the product by its extreme fluidity and dustlessness.

One important field of use for our invention is the carbon black industry, for example, in connection with the so-called "channel" carbon black produced by burning natural gas flames against metallic surfaces. The ultimate carbon black particle is so small that its shape and exact size are beyond microscope determination. As first produced, such carbon black has an apparent density in bulk of about three pounds per cubic foot. It has been the practice to increase this apparent density in bulk to about twelve pounds per cubic foot by stirring and then to increase it further to about twenty-five pounds per cubic foot by compressing. The resulting compressed carbon black due to its dusty nature is still inconvenient to handle because it so readily reverts to its dusty form. In addition to this, the difficulties of packaging carbon black by compression have necessitated the employment of small sacks, twenty-five pounds or less in capacity, and for practical reasons the industry has been obliged to standardize on two sizes of sacks.

We have found that by subjecting the carbon black of commerce to certain conditions of turbulent pressure, the fine dusty particles of which it is composed may be caused to adhere and agglomerate firmly with each other in groups. Thus the carbon black may be coverted into small spherical granules of apparent density in bulk higher than before such treatment, of relatively tenacious structure, with each of such granules presenting a more or less polished, non-adherent surface. Carbon black in this spherical grain form possesses all of the advantages of convenience in handling and distribution above discussed and, in addition, it disperses perfectly in rubber mixtures, and is otherwise as useful or more useful in rubber making and other industries than the compressed carbon black of commerce formerly employed.

Similarly we have found that a number of other fine, dry powders, among them, all of the carbon pigments commonly known as lamp blacks, zinc oxide, iron oxide and clay, (previously reduced to the requisite state of sub-division if necessary) have the property of agglomerating under determinable conditions of mechanical manipulation and that when subjected to suitable turbulent pressure they may be converted into small, tenacious spherical granules of concentric shelled structure and having non-adherent surfaces.

The powders which display this behaviour have certain other characteristics which serve to differentiate them from those powders which cannot be agglomerated by such treatment. For example, if jigged on a vibrating surface, they will agglomerate into small spheres of very fragile structure, they will form an adhesive lump when squeezed, they will adhere to metallic or non-metallic rods when these are forcibly drawn through them and they are free from gummy and resinous ingredients. The present invention has to do with fine powders or pigments having these properties or characteristics and for convenience they will be termed herein as "agglomerative powders". The powders listed above are typical but this list is far from complete and those mentioned are named as illustrating but not limiting our claims for this invention as applying to all agglomerative powders.

We have discovered that by subjecting them to the proper conditions of turbulent pressure

and to proper manipulation, these agglomerative powders may be converted into granules of greatly increased apparent density, having in many instances internal structures of spherical shells concentrically disposed about central cores, with each granule presenting a smooth, polished, non-adherent and non-coherent exterior surface. The self-generated spherical granules thus produced are of appreciable mechanical strength and, since they have the property of withstanding very considerable direct pressure, they may be handled or transported in bulk with practically no disintegration. The powder, converted into this spherical-grain form, is substantially dustless, free-flowing and non-adherent.

The proper conditions of turbulent pressure necessary for the conversion of agglomerative powders into such spherical granules may be created by the use of various types of mechanical apparatus. Examples of suitable apparatus are disclosed in our pending applications Serial Nos. 642,850 and 40,438. We have found that, under certain conditions, we can directly convert fine agglomerative powders from their loose, seemingly amorphous form into this dense, spherical-grain form. However the process is usually more rapid, economical and commercially efficient if a priming charge of pre-formed spherical material is mixed with the powder being treated. This priming material may be either the same or different from the powder. If such a priming charge is not used, the first effect of turbulent pressure on the powder may be to cause the gradual formation of a small number of nuclei consisting of a number of powder particles adherently bonded together. Since, in an adequate space of time under turbulent pressure, the nuclei as they form are pressed upon from all sides and in all directions, their growth is in three equal dimensions, thus their form is spherical.

The priming charge serves at least two important purposes, i. e. (1) It immediately provides cores or nuclei upon which the powder may at once begin to adhere, thus promoting or expediting formation of a large proportion of spherical, well-agglomerated granules of low viscosity. The resulting lower average viscosity enhances the turbulent effect of the mechanical apparatus used and thereby accelerates the conversion from powder to dustless spherical agglomerate. (2) It provides immediately bodies of much larger weight than the weight of the particles of dust. These larger bodies, by collision, provide impacts on the dust particles of sufficient intensity to cause permanent adhesion. They also widen the zones of effective action in the apparatus and so permit the use of greater clearances than would otherwise be the case, thereby simplifying the mechanical construction of the apparatus.

There are a number of ways of producing satisfactory priming charge material but all are alternative methods for accomplishing two purposes; first, exerting sufficient pressure on the powder particles to form firm adhesions; second, reduction of the agglomerates thus formed to a convenient and suitable size and with three appreciable dimensions. For example, satisfactory cores may be secured from some agglomerative powders by subjecting relatively thin layers of the powders to direct compression, sufficiently heavy to cause the formation of a chunk, cake or briquette. This, if then rubbed through a wire screen of suitable mesh, is partially disintegrated and the resulting product of this treatment is suitable for core material and may be readily built up into spherical form by the addition of concentric external layers or shells.

As another example, spheres of foreign material of suitable density and size, may be mixed with the agglomerative powder and the mixture subjected to turbulent pressure. The powder will then form a substantially concentric, spherical shell on each sphere. If the composite spherical granules thus formed are rubbed upon a screen of the proper mesh to exclude the original spheres, they will break and fragments of the shells passing through the screen will be found sufficiently dense and adhesive to serve as priming material for the subsequent agglomeration of powders of their own nature. Examples of spheres suitable for this purpose are fine shot or seeds, such as those of the beet, clover or petunia.

Likewise, composite granules may be formed by using cores made from one powder with shells formed from an entirely different powder for use where a free-flowing dustless mixture of two or more such powders is commercially desired.

An important feature of our invention lies in our discovery of the fact that we are able to take these small agglomerates, which may be so fine as to partake of the characteristics of dust, and build them, through successive addition of shells, to a size that imparts to the product the commercial advantages above discussed, viz., dustlessness, high density, low mass viscosity, spherical shape and non-adherent surface.

Another important feature of our invention lies in our discovery of the further fact that we can alter and control the degree of surface polish imparted to the granules since, under proper conditions, this is a function of time of treatment.

Our ability to control the degree of surface polish enables us:

1. To control the viscosity of the material in bulk, thus facilitating handling.

2. To offset the increase in viscosity in bulk which would otherwise naturally accompany increase in average diameter of the individual spheres.

Thus, ability to control polish enables us to maintain adequate turbulent pressures throughout a wide range of average sphere diameter and thus to add a greater number of concentric shells and produce larger and more useful spherical agglomerates.

Since the impacts delivered by one sphere to another are a function of the weight of the spheres, it becomes possible to increase rapidly the density of the spherical granules in process of treatment by employing a priming charge of large spheres. We may, by proper manipulation and adequate time of treatment, thus cause the density of the granules to approach the absolute specific gravity of the material being treated. This is another characteristic and extremely important feature of our invention.

For purposes of illustration, the invention will now be more specifically described as carried out in connection with the production of spherical grain carbon black. In the accompanying drawing,

Fig. 1 represents spherical grain carbon black as seen in the field of a microscope, magnified 80 diameters;

Fig. 2 represents spherical grain zinc oxide, the granules being shown in cross section in the field of a microscope, magnified 80 diameters;

Figs. 3 and 4 are sectional views of single spherical granules, magnified 300 diameters; and

Figs. 5 and 6 represent diagrammatically the conversion process as carried out by one form of suitable apparatus.

In the preparation of spherical grain carbon black from the carbon black of commerce, the cores may be first produced separately by subjecting the seemingly amorphous powder to prolonged treatment under conditions of adequate turbulent pressure. The cores thus produced present a smooth, non-adherent surface and may conveniently have an apparent density in bulk of more than 12 pounds per cubic foot, for example, about 20 pounds per cubic foot. The average size of these cores, as measured by sieve tests, is determined by the degrees of turbulence and pressure to which the powder is subjected. The optimum size is determined by the number of concentric shell-like layers which are subsequently to be added to the core and by the design of the apparatus in which the additions are made. For carbon black, an average core diameter ranging from 300 mesh to 60 mesh has been found satisfactory.

Substantially equal amounts of these cores and of commercial carbon black may now be introduced into an apparatus such, for example, as that disclosed in our pending application Serial No. 642,850 or 40,438. This apparatus comprises a drum which constitutes a container and in which are arranged a plurality of impelling elements and baffle elements movable past each other in an approaching and receding manner and provided with means to cause also an up-and-down circulation in the mass delivered thereto. The composite charge may be thus subjected to pressure and to turbulence causing this pressure to be multi-directional upon each particle thereof. This is accomplished by a sliding or shifting action of the mass upon itself under conditions of turbulence and pressure and a stroking or polishing interaction of its particles. Under these conditions there occurs an agglomeration of the flocculent carbon black upon the cores or fines with the result that a spherical shell is built up upon each core.

In Fig. 4 of the drawing is shown a core 10 upon which has been formed a concentric shell 11. This figure represents a single granule of spherical-grain carbon black at high magnification, the core being shown in elevation and the concentric shells in section.

The process may be continued until all of the flocculent carbon black has been agglomerated upon the cores in the form of spherical shells and if the components of the initial charges are substantially equal, it is apparent that each shell will comprise substantially 50% of the weight of the spherical grain thus formed. It is desirable to continue the process for a time after the disappearance of the flocculent carbon black from the apparatus sufficient to polish the surfaces of the spherical grains. At this stage the entire charge has been converted to spherical granules of high apparent density, the individual granules being sufficiently tenacious to stand such pressures as they may be subjected to in handling, and presenting a more or less polished non-adherent surface. If the process is continued further, the granules tend to become more and more dense and to be reduced in diameter. The process may be stopped when the spherical granules have thus reached a convenient commercial density and polish.

We have described the production of spherical granules comprising a core and a single concentric shell by a single regenerative process terminating when an equal weight of flocculent carbon black has been agglomerated upon each core. If desired, a new charge of flocculent carbon black may now be introduced into the apparatus and, as before, this may approximately equal in weight the amount of the spherical grain charge already produced or remaining therein. The process is now renewed as before and a second concentric shell agglomerated upon the outer surface of the first. Such a second shell is indicated by reference character 12 in Fig. 4 of the drawing. When the flocculent carbon black of the charge has again agglomerated, the process may be repeated. In Fig. 4 a third concentric shell 13 is shown and in Fig. 2 are shown granules having as many as six concentric shells agglomerated about the core. What has already been said applies to these additional regenerative processes. The core and each shell is defined by a polished or somewhat glazed spherical surface upon which the next larger shell is agglomerated and to which it directly coheres. These surfaces may be readily detected as surfaces of cleavage or division in partially crushed or sectioned spherical granules.

Fig. 1 represents spherical-grain carbon black or of other agglomerative powders as seen in the field of a microscope in the magnification of 50 diameters. It will be noted that all the grains are spherical or substantially so and that there is a substantially uniform upper limit of diameter which has been attained by a large number of the granules. The smaller granules represent those which contain in their composition a smaller number of concentric shells than the larger granules, or the shells of which are thinner.

We have found that in the case of carbon black subjected to treatment in the type of apparatus described in our pending application Serial No. 642,850 or 40,438, it is best to use a priming charge not coarser than 60 mesh if an increment of equal volume is to be added in the form of a shell, because the resulting spherical grains produced by such an increment will then have a size of about 40 mesh and using carbon black, we have found that the subjection to turbulent pressure of a charge of spherical grain carbon black containing a substantial percentage of grains substantially larger than 40 mesh in the type of apparatus described, results in viscosities and pressures so great as to crack and burst the granular agglomerates, thus impairing the free-flowing and dustless characteristics of the charge. For this reason, when treating carbon black by the above process, we have found it advantageous to start with a priming charge in the range of 150 mesh so that a large number of concentric shells could be added before reaching the limiting size.

As the diameter of the spherical grains increases with the addition to them of successive increments of carbon black, and also as the application of turbulent pressure is continued after all the flocculent carbon black has been applied to the cores of the priming charge, some of the shells become ruptured into fragments but not into dust, and these fragments, in the presence of their neighbors, round off and become to all intents and purposes new core material. This process continues throughout the run and results in a decrease of the average size of the agglomerates with time of running. It is also to be

understood that it is the natural tendency of all these agglomerates to become smaller with time of running because of increase in density due to the impacts and pressures applied to their surfaces.

At the conclusion of the first cycle of the operation, the finished charge may be withdrawn from the apparatus and in this condition constitutes a satisfactory and valuable commercial product. It is usually preferable, however, to sift the material at the end of each cycle, separating the smaller agglomerates from the mass so that they may be used as priming material for new charges, and retaining in the finished product only agglomerates of the larger sizes.

In the light of our present knowledge, we may suggest one theory of what takes place within the drum to cause the carbon black to assume its new and relatively dense form by reference to Figs. 5 and 6 of the drawing. The stationary elements 42 are arranged alternately with the moving elements 32 and the charge of black surrounds and entirely fills the space between these elements. As the elements 32 move relative to the elements 42 (Fig. 5), they tend to carry the black in a mass therewith but such movement of the black is opposed by the stationary elements 42. The result is that cones 86 and 87 of black form both forwardly and rearwardly of the elements 32 and move along with these elements, while cones 88 and 89 of black form both forwardly and rearwardly of the elements 42 and remain stationary therewith. The carbon black intermediate the elements 32 and 42 (indicated by line 90 of black particles in Fig. 5) is in a state of turbulence, the black adjacent to the elements 32 moving nearly as fast as those elements and the carbon black adjacent to the elements 42 remaining nearly stationary. The relative positions of the elements are constantly changing as the elements 32 approach and recede from the elements 42 and, due to this action, the particles of carbon black are alternatively being brought into most intimate and bombarding relation whereby they are compacted into the relatively dense form of the finished product and into a relatively loose relation, wherein they are free to rearrange themselves for the next impact. Thus the black particles are intermittently subjected to pressures or impacts and are relatively rearranged between the successive impacts, such impacts or pressures thereby becoming multi-directional, i. e., not occurring twice in succession in the same direction.

If the cores are formed from compressed or briquetted material, we have found it advantageous to subject the rough fragments resulting from this method to turbulent pressure for a short period. This treatment breaks off the rough irregular projections of such cores, tends to equalize the three dimensions, fills in the cavities with the detritus resulting from the abrasion of the projections and thus produces cores which are more nearly perfect spheres. This condition is desirable because the more nearly spherical the priming charge, the lower the viscosity of the subsequent mixture with the agglomerative powder. Lower viscosity, within limits, enhances the turbulent pressure effects of our apparatus and thus assists the conversion process. If the core is spherical to start with, the shell agglomerated thereon will also be spherical. If the core is irregular in its shape, the shell will not be perfectly spherical but will tend to approach spherical shape more nearly than the core. In Fig. 3 is shown a core 18 of more or less irregular shape, such as might be found by the compressing and breaking process, and upon this has been formed a shell 18. This, it will be noted, tends to fill in and smooth out the surface irregularities of the core and to approach spherical shape.

Fig. 2 represents spherical granules of zinc oxide which have been imbedded in a transparent medium and then sectioned, as seen in the field of a microscope in the magnification of 60 diameters. In this view, the cores 20 of certain granules are shown in place whereas other granules 22 are shown as having lost their cores. These were displaced and lost in the operation of sectioning the spherical granules. As above suggested, the cores 20 may be of zinc oxide or they may be of carbon black or other agglomerative powder.

The limiting size of the agglomerates depends somewhat upon the design and operation of the agglomerating apparatus used for producing the requisite turbulent pressures, but is even more dependent upon the nature of the agglomerative powder being treated. Zinc oxide, for example, can be caused to agglomerate by increments into spheres of larger than 20 mesh before the resulting increased pressure exceeds the structural resistance of the spherical grains and break down into dust ensues.

The continuous tendency toward the replacement of fine material as the process is carried out is advantageous for two reasons:

1. It maintains the distribution of particle sizes.

2. It affords a continuous replacement of fine material for the start of new cycles if these fine particles are screened out at the end of each run, thus removing the necessity for a separate and distinct process or step for the production of cores.

The distribution of particle sizes is important to the process for three reasons:

1. It decreases the viscosity of the charge by providing supporting surfaces in the interstices between the larger particles, thus increasing the ability of the apparatus to produce adequate turbulent pressures.

2. It decreases the liability of the material to be ruptured or crushed by conveying, transportation, or handling, thus inhibiting the premature return of the spherical grains to their original, dusty fine components.

3. It increases the apparent density in bulk of the commercial product over that which would obtain if only one size of particle were present, thus economizing on packing materials and storage space per unit weight.

While in describing the process of our invention as carried out in connection with carbon black, we have suggested the addition of an equal weight of flocculent material to the priming charge, it is entirely practicable to double this proportion, that is, to add flocculent carbon black in an amount equal to twice the weight of the priming charge. The percentage of priming material necessary or advisable in the charge is a function of the combined viscosities of the priming material and the fine powder. From this it follows that it is possible to use a 5% or 10% priming charge in treating certain powders, whereas a 33% priming charge may be required in treating other fine powders. In the case of carbon black, a 33% priming charge is the minimum below which it is not convenient to go.

This application is a division of our application Serial No. 694,894, filed August 12, 1933 which

issued as Patent No. 1,997,214, which in turn was a continuation-in-part of our earlier application Serial No. 622,104, filed July 18, 1932, and corresponding to our Canadian Patent No. 323,741, granted July 4,1933. Patent No. 1,997,214 has now been reissued as Reissue Patent No. 19,780, November 12, 1935.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. As a new product of manufacture, polished spherical granules each comprising a tenacious core of one agglomerative powder enclosed in a plurality of concentric shells of another agglomerative powder, all united in an integral body of sufficient tenacity to withstand disintegration when handled in bulk.

2. As a new product of manufacture, polished spherical granules comprising in their structure a tenacious core and a concentric shell of fine dry agglomerative powder united thereto with sufficient tenacity to withstand disintegration when handled in bulk, and of the core and shell one being zinc oxide and the other lamp black.

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March 5, 1940. H. J. GLAXNER Re. 21,379

METHOD OF MAKING AGGREGATES OF CARBON BLACK

Original Filed Nov. 6, 1933

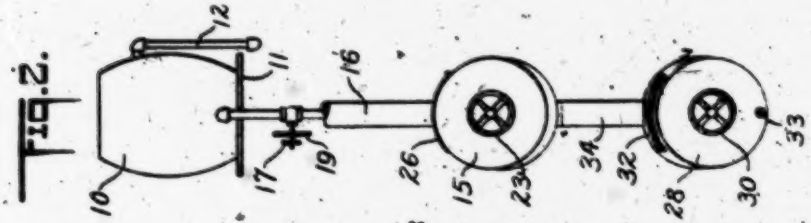


Fig. 2.

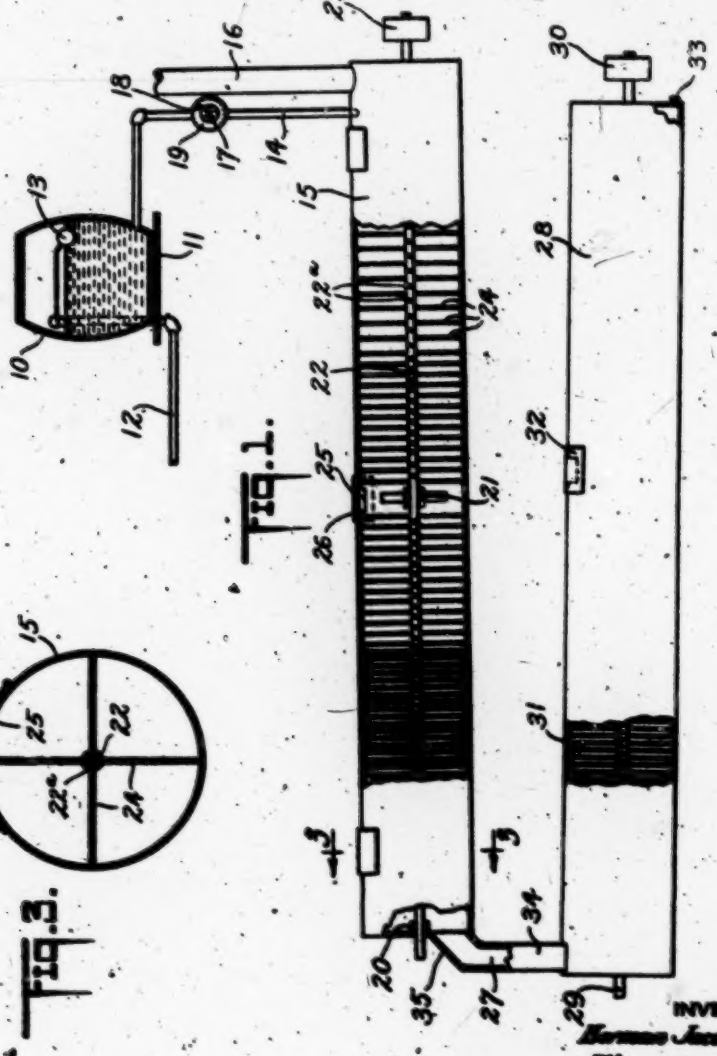


Fig. 1.



Fig. 3.

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Re. 21,379

UNITED STATES PATENT OFFICE

21,379

METHOD OF MAKING AGGREGATES OF CARBON BLACK

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Original No. 2,065,571, dated December 22, 1936, Serial No. 696,774, November 6, 1933. Application for reissue August 2, 1939, Serial No. 288,176

1 Claim. (Cl. 134—99)

The term carbon black is used herein in its broader sense to include lamp black, gas black and other black analogous carbon substitutes commonly employed as pigments or fillers. Furthermore, the apparatus and process may be successfully utilized in treating other finely divided material of a flocculent nature and particularly for transforming an impalpable powder into relatively minute granules or dustless aggregate so as better to adapt it for purposes of handling, shipment or storage and use.

Certain characteristics of carbon black which render it particularly valuable in the manufacture of rubber compositions, phonograph records, various wax and resin compositions, printers' ink and other preparations are its extremely fine state of sub-division together with its capability of being uniformly dispersed in rubber, waxes, oils and the like.

Carbon black as manufactured is such a very light, fine powder that normally ten pounds or less occupy a package of one cubic foot. Such light, bulky material is expensive to pack, ship and store, and handling the light powder causes it to fly in the air to such an extent that there is not only substantial loss of material but great annoyance to workmen breathing the dusty air.

An object of the present invention is to so treat the carbon black that it is decreased in bulk for purposes of shipment, storage and handling and so that it is substantially dustless in handling and yet capable of ready and uniform dispersion.

It has heretofore been proposed to accomplish these results by the formation of aggregates or pellets of carbon black substantially free from the occluded gases which cause the powder to be so bulky, the pellets themselves being readily crushable to return the carbon to its original pulverulent condition.

Insofar as the process is concerned, an object of the invention is to simplify the process of manufacturing the carbon black aggregates by avoiding the need for using two immiscible liquids and by providing a process involving merely the addition of water to the black, mechanical agitation and drying. No heat treatment or chemical agent is used in forming the dustless granules and drying of the granules may be accomplished in any conventional manner. No screening, filtering, decanting or other separating step is necessary.

A further object is to provide a process of manufacturing the carbon black requiring no delicacy of technique and capable of perfect control simply by varying the speed of the agitation and the percentage of the liquid which is mixed with the black.

Insofar as the apparatus is concerned, an object of the invention is to provide an extremely simple apparatus consisting merely of agitating means and means for feeding the carbon black and liquid into it and automatically operative to transform the carbon black and liquid into minute granular aggregates which need only to be dried to produce the finished product.

The invention may be more fully understood from the following description in connection with the accompanying drawing, wherein:—

Fig. 1 is a somewhat diagrammatic view of an apparatus embodying the invention, parts of this apparatus being shown in section and parts in elevation,

Fig. 2 is an end elevational view of the apparatus shown in Fig. 1, and

Fig. 3 is an enlarged transverse sectional view on the line 3—3 of Fig. 1.

Referring with more particularity to the drawing, 10 represents a reservoir mounted upon a suitable support 11 and into which a suitable liquid such as water flows from a pipe 12, the water in the reservoir being kept at a constant level by the float valve 13. Water drains by gravity from the reservoir 10 through a pipe 14 into the inlet end of a cylinder or tube 15. The carbon black is delivered into the inlet end of this cylinder through a pipe 16. Any suitable means (not shown) may be used for delivering carbon black at a constant rate through pipe 16 and the amount of water which flows into the cylinder to mix with the carbon black may be controlled by a needle valve 17 in the pipe 14. The handle of this valve preferably carries a pointer 18 reading upon a fixed scale 19 associating with the valve stem so that the setting of the valve is always visually indicated and micrometric adjustments of the valve may be made.

Cylinder 15 which is closed at both ends inclines slightly downwardly from its inlet to its outlet end. Bearings such as 20 at the ends of the cylinder cooperate with a central bearing 21 to support a shaft 22, the axis of which coincides with the longitudinal axis of the cylinder. The projecting end of the shaft 22 carries a pulley 23 or other suitable power receiving means.

A plurality of agitator arms 24 are fixed upon the shaft 22. As seen in Fig. 3 these arms extend through and are fixed within transverse openings 25 in the shaft 22, each arm projecting

equal distances from opposite sides of the shaft and having its ends terminating just short of the wall of the cylinder. Alternate arms are arranged at right angles to each other.

The arms occupying approximately that third of the cylinder adjacent the inlet end of the latter are spaced somewhat farther apart than the arms occupying the central third of the cylinder and those arms occupying the third of the cylinder toward the outlet end are spaced even a less distance apart.

At intervals throughout its length, the stationary inclined cylinder 16 may have a number of inspection openings 26 in its top, these openings being normally closed by pivoted cover plates 26. They are designed to permit observation of the process which is going on in the cylinder 16 and the central closure 26 may be opened to provide access to the central bearing 21.

Material from the outlet end of the cylinder 16 passes through a pipe 27 into the inlet end of a substantially horizontally disposed cylinder 28 arranged beneath the cylinder 16. The two cylinders are of generally similar construction, cylinder 28 having a central shaft 29 corresponding to shaft 22 and a driving pulley 30 corresponding with pulley 23. The shaft 29 likewise carries agitator arms 31 corresponding to the arms 24 but arranged at the same distance apart as the arms in the lower third of the cylinder 16. Cylinder 28 may be provided with an opening and with a trap door 32 through which the central bearing thereof may be inspected.

The outlet from the cylinder 28 is through a relatively small discharge passage 33. The pipe through which material passes by gravity from the upper to the lower cylinder includes a vertical portion 34 discharging into the lower cylinder and an angularly disposed portion 35 disposed at approximately a 45° angle to the longitudinal axis of the upper cylinder and communicating with such cylinder below the shaft bearing 21.

Before explaining the operation of the apparatus or the method of carrying out and controlling the process, it may be noted that in a typically commercial installation which he has found to be satisfactory for the purpose, each cylinder may be about eighteen feet long and about thirty inches in diameter. The agitator arms near the inlet end of the upper cylinder 16 are arranged on approximately 2" centers and the arms themselves are about ⅜ of an inch in diameter. At the central third of the cylinder the arms are set on about 1½" centers and at the lower third of the cylinder 16 and throughout the length of the cylinder 28, the arms may be arranged on 1" centers. The agitator arms 24 are of proper diameter to fit snugly within the openings in their supporting shaft and a spot weld on each arm may be made just against the shaft to keep the arm from slipping after it has been driven through the shaft to an extent where it projects an equal distance from opposite sides thereof.

It may be noted here that it is within the scope of the invention to use one long cylinder instead of two shorter ones but for practical purposes it is undesirable to impose too great a torque on the relatively light shaft which carries the agitator arms. Accordingly he prefers to use the two cylinders and shafts and driven pulleys. The operation of the device is substantially as follows:—

With the shafts of both cylinders rotating (a speed of about 70 R. P. M. has proven satisfactory), the carbon black and water are admitted into the inlet end of cylinder 16. The water, incidentally, may be chemically pure but commercially pure water is in general satisfactory.

The water and the carbon black form a relatively heavy doughy mass in the higher or inlet end of the upper tube 16. By observation it can be readily discerned whether this dough is of proper consistency. The valve 17 can be accordingly adjusted until the desired consistency of the doughy mass is obtained. In practice the admission of carbon black and water in about equal amounts of weight has proven very satisfactory.

By the time the continuously agitated mass has reached the middle of the tube 16, the carbon black should be just a little more moist than a lump of carbon black formed by a drop of water falling into loose carbon. If the central inspection door of the tube 16 be opened and a small lump of the wetted carbon black removed, the lump should be almost stiff enough to break instead of being of a plastic and sticky consistency.

By the time the wet carbon black gets to the outlet tube 27, part of it is already in the form of pellets or granules free from occluded gases and the work is completed in the cylinder 28. The outlet opening of the cylinder 16 is of such a size and so positioned as to prevent too rapid passage of material from the tube 16 into the tube 28 and thereby avoiding overburdening the shaft and agitators of the latter tube or cylinder.

So far as he has been able to determine by observation, the discharge of carbon black from the cylinder 16 is somewhat as follows:—

The agitator arms 24 on the shaft 22 run close to the end of the tube and have a tendency to throw the material sideways. This conclusion is drawn from the fact that the arms being round or packed up to an edge with dough, the dough balls are knocked out of the path of the arms by the arms themselves, otherwise the dough would be ahead of the arms and would have no way of freeing itself from adhering to the pin. This adhesion does occur to a certain extent until a wedge shape formation of dough is built up to a sharp edge on the front side of each arm. As this dough is forced sideways from the arm next to the outlet end of the cylinder, some of it hits the outlet end and is allowed to enter the lower cylinder through the connecting pipe. This gives about the proper restriction to prevent too rapid release of the material being worked upon, into the lower cylinder. The speed of release may also be controlled to some extent by the inclination of the upper cylinder. However if this inclination is too steep it allows an accumulation of dough at the outlet end and causes the agitator arms adjacent said end to do most of the work. By a correct inclination, the work of the agitator arms will be equalized throughout the length of the tube and he has found that about 3 or 4 inches drop in 18 feet is an inclination which works out successfully with proper distribution of the work to all of the agitator arms, assuming that the proper mixture is being fed into the upper end of the cylinder.

For a while after the machine is started, part of the doughy mass sticks to the cylinder, forming a thin coat of paste all around it. As this coat builds up, the pins near it cut it loose although it builds slightly more between the path of the pins. In operation, aside from this thin coating near the inlet end of the upper cylinder,

the mixture is capable of movement at all times since the pins are set so close together and travel at such speed that the lumps and pellets which they form are kept flying at all times.

The action which takes place in the cylinder 28 is similar to that which takes place in the cylinder 18 except that it completes the operation, reducing the moist carbon black to small granular sized, spherical aggregates. While the cylinder 28 might be slightly inclined if desired, it operates successfully even though horizontal, apparently for the reason that as the dough forms into pellets or granules, they have a tendency to travel faster and easier. As these granules are forced out of the outlet of the cylinder 28 they are of substantially uniform spherical shape and of substantially uniform size (typically) in the order of .025 of an inch in diameter.

The granular spherical aggregates delivered from the cylinder 28 may be dried in any conventional manner and are then ready for packaging and shipment.

The process is characterized by its complete lack of any complex technique and by the ease with which the uniformity of the finished product may be controlled. For any given amount of carbon black introduced continuously into the pipe 16 a roughly corresponding amount by weight of water should be introduced through the pipe 14. The inspection windows in the cylinder 18 make it readily apparent if improper proportions are being introduced and so that the necessary micromatic adjustment of the valve controlling the water flow may be made. With cylinders of definite length and with the proper proportions of water and carbon black entering the upper cylinder, the only variable factor is the speed of operation of the agitator shafts and he has discovered that by increasing or decreasing the speed of these shafts either larger or smaller spherical aggregates may be formed.

Even careful observation of the process has not yet enabled him to be dogmatic in advancing an explanation of the reasons for the production of substantially spherical uniform sized aggregates, but he may advance the tentative theory that substantially every physical contact which is had between the moist carbon black lump aggregates and the walls of the cylinders or the agitator arms, is productive of a balling effect, that is to say, whether the lumps of wet carbon are struck by the arms which are of circular shape or rolled down the surfaces of the cylinder, all of the physical contacts of the apparatus with such aggregates and also the combination of all such contacts apparently tends to roll them into substantially spherical shape. The size of the spheres produced is apparently controlled to some extent by the spacing of the agitator arms and to some extent by the speed of agitation.

Having thus described his invention, what he claims as new and desires to secure by Letters Patent is:—

He claims:

The method of making generally spherical, relatively minute aggregates of carbon black, which are in the order of .025 of an inch in diameter, and substantially devoid of occluded gases, which includes the steps of mixing approximately equal quantities of water and carbon black to form a pasty mass, and progressively advancing the mass through an elongated path, and simultaneously forming said aggregates by subjecting the advancing mass to the action of a multiplicity of impacts caused by agitators of small cross-sectional area, spaced apart at comparatively small distances, and movable transversely of the direction of advancing movement of said mass.

COLUMBIAN CARBON COMPANY,
Assignee of Herman Jacob Glasner, Deceased,
By REID L. CARR,
Secretary and Vice President.

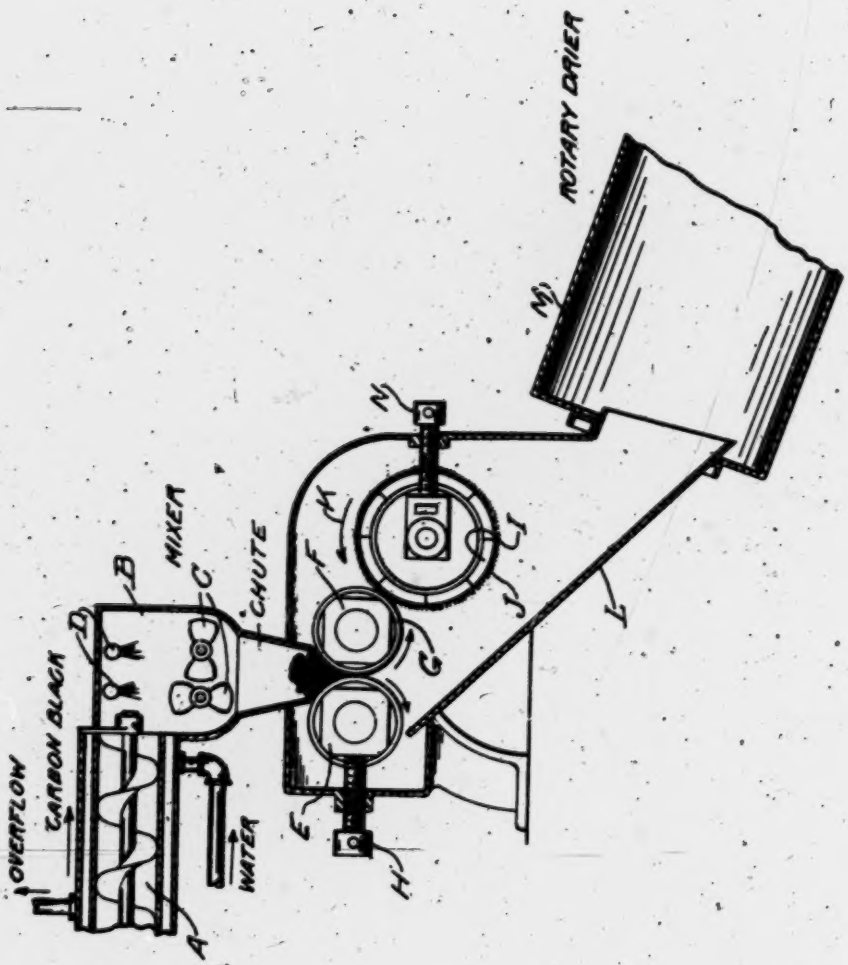
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March 12, 1940. H. W. GROTE Re. 21,390

PROCESS OF FORMING AGGLOMERATED CARBON-BLACK

Original Filed Aug. 22, 1933.



INVENTOR.
H. W. Grote
BY Foster & Codier
ATTORNEYS.

Reissued Mar. 12, 1940 Re. 21,390

UNITED STATES PATENT OFFICE

21,390

PROCESS OF FORMING AGGLOMERATEL CARBON BLACK

Hans Walter Grote, New York, N. Y., assignor to United Carbon Company, Inc., Baltimore, Md., a corporation of Maryland

Original No. 2,040,770, dated May 12, 1936, Serial No. 686,311, August 22, 1933. Application for reissue September 27, 1937, Serial No. 165,943

10 Claims. (Cl. 134—60)

The present invention relates to the production of agglomerates or shaped masses of carbon black, for the purpose of overcoming some of the objectionable properties of raw carbon black, such as the formation of dust, during the handling of the same, during the mixing of the same with rubber or other materials, and for producing a product which is easier to handle. As is well known, carbon black in its original form is a powdery or more or less lumpy material constituted of extremely small particles, so that during the handling of the same, air currents may blow the same about to some extent, and the object of the present invention is to produce agglomerates which will be free from the said objection.

In accordance with the present invention, the carbon black is moistened with a volatile liquid, e. g., alcohol, benzol, gasoline, methanol and acetone, but for which purpose water is entirely suitable, the mass is stirred and kneaded, to form a rather stiff paste, which is friable in character. The said paste is then worked upon an ordinary roller mill, for example a mill consisting of two horizontal rollers rotating in a vertical plane, the rollers may be operated at slightly different surface speeds, in order to cause the roll-milled material to adhere to one of the rollers, as a compacted layer. When this has been accomplished, another roller carrying needles or other points of similar character is rotated in such a position that the needles will scratch off the material on the roller, thereby producing shaped masses of the carbon black, which are then subjected to a high temperature drying operation, preferably at a temperature very much above the boiling point of the wetting agent employed.

The dried material so produced can then be passed over a series of sieves in order to separate the same into different sizes, as required for different particular purposes for which the carbon black is to be employed.

For carrying out the process of the present application, an apparatus can be employed such as is shown diagrammatically in the annexed drawing, the figure representing a sectional elevation of suitable apparatus. It is to be understood however that this apparatus is shown merely for the purpose of illustration, and not as limiting the scope of the invention thereto.

In carrying out the process of the present invention, the carbon black may be fed through a conveyor A, which may be water-jacketed if desired for cooling purposes, the carbon black falling into the mixer B which may be a trough-shaped device, having agitators also acting as propellers C. Jets of a suitable vapor or liquid or mist are directed upon the material from the spayers or perforated pipes D, and the proportion of the liquid used should be such as to give a very friable relatively dry pasty mass, in the mixer B.

From the bottom of the mixer B, the pasty mass is discharged through the chute illustrated, or in any other manner, into the space between the rolls of a roller mill, shown at E and F. These rolls rotate, in the direction of the arrows shown, the peripheral speed of one roller may be slightly greater than that of the other so that the moistened carbon forms in a layer G, upon the roller F. The thickness of this layer will be regulated by the space between the two rollers, this being adjustable by means of a screw shown conventionally at H.

At I is shown a rapidly rotating roller, which can rotate for example in the direction shown by the arrow K, the surface of the roller carrying a great number of small needle-shaped spikes J. These needles, which are distributed over the entire face of the roller I, scratch off the layer or cake of compressed moistened carbon from the surface of the roller F, this then flowing down through the chute L, into the upper end of a rotary drier M, in which the material is dried by being subjected to gases at a high temperature, say 400 to 500° F. in order to rapidly dry the same, and leave the desirable porous friable structure of the finished material.

The roller I should be situated near enough to the roller F so that the needles will scrape off substantially the whole of the layer of carbon therefrom, the position of said roller being adjusted by means of a screw shown at N, or otherwise. The closeness together of the needles J will regulate the size of the particles torn off in this manner, which particles will of course be of more or less irregular shape.

After the drying operation the granular material can be passed over a series of sieves, to separate the same into different particle sizes, and any particles which are too small for use, as well as any particles which are too large for use, can be reintroduced through the conveyer A into the mixer B.

The particle size will be in part regulated by the thickness of the layer G on the roller F, and for ordinary purposes I find that this layer can be from a sixteenth of an inch in thickness to a quarter of an inch, but I do not restrict myself to these limits. If any of the moistened carbon is left on the roller F, after passing the roller I,

this does no particular harm and will bond with a subsequently applied layer of the paste, to be scratched off during a subsequent rotation of the roller.

The proportion of water or other liquid introduced into the carbon black in the mixer B is subject to variation, and for ordinary purposes I find that about 40% of moisture is very satisfactory. This figure however is given merely by way of illustration without restricting the invention thereto.

I claim:

1. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening carbon black with a volatile liquid, forming a thin sheet of the moistened carbon black, subdividing said sheet into small irregularly shaped masses of carbon black agglomerates and drying the said masses, whereby a granular product is formed which is substantially dustless.

2. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening the carbon black with water to form a stiff, pasty mass, forming a compressed sheet of the moistened carbon black, subdividing said sheet into small irregularly shaped masses of carbon black agglomerates and drying the said masses, whereby a granular product is formed which is substantially dustless.

3. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening carbon black with water, the amount of water being about 40% of the moistened mass, forming a compressed thin sheet of the moistened carbon black, subdividing said sheet into small irregularly shaped masses of carbon black agglomerates and drying the said masses, whereby a granular product is formed which is substantially dustless.

4. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening carbon black with a volatile liquid, forming a thin sheet of the moistened carbon black, subdividing said sheet into small irregularly shaped masses of carbon black agglomerates, and subjecting the said masses to temperatures within the range of approximately 400° to 500° F. while agitating the masses by a rotary tumbling motion to thereby dry the masses as shaped porous, friable granules of substantially dustless carbon black.

5. The process of converting finely divided and dusty carbon black into a product which is substantialy dustless, comprising moistening the carbon black with water to form a stiff, pasty mass, forming a compressed sheet of the moistened carbon black, subdividing said sheet into small irregularly shaped masses of carbon black agglomerates, and subjecting the said masses to temperatures within the range of approximately 400° to 500° F. while agitating the masses by a rotary tumbling motion to thereby dry the masses as shaped porous, friable granules of substantially dustless carbon black.

6. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening carbon black with a volatile non-solvent wetting agent and working the mass to form a friable relatively dry paste, subjecting the wetted mass to roll milling to form said paste into a compacted layer of wetted carbon black agglomerates, breaking said layer to form small, irregularly shaped agglomerates therefrom, and thereafter drying the said small irregularly shaped agglomerates at temperatures within the range of approximately 400° to 500° F., whereby substantially dry, porous, friable granules of a substantially dustless product are formed.

7. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening the carbon black with water and working the mass to form a stiff paste, subjecting said paste to rolling pressures by a pressure-exerting roller member to compress the mass and form a sheet-like compacted layer therefrom, breaking said compacted sheet-like layer into small shaped masses of wetted carbon black agglomerates, and thereafter drying the said small shaped masses of wetted carbon black at temperatures within the range of approximately 400° to 500° F. while agitating the masses by a rotary tumbling motion, whereby a substantially dry, porous, friable granular product is formed which is substantially dustless.

8. The process of converting finely divided and dusty carbon black into a product which is substantially dustless, comprising moistening-carbon black with approximately 40% by weight of water and working the mass, subjecting the moistened carbon black to rolling pressures by a pressure-exerting roller member to compress the mass and form a sheet-like compacted layer therefrom, breaking said compacted sheet-like layer into small shaped masses of wetted carbon black agglomerates, and thereafter drying the said masses while agitating the masses by a rotary tumbling motion at temperatures within the range of approximately 400° to 500° F., whereby a substantially dry, porous, friable granular product is formed which is substantially dustless.

9. The method of making minute aggregates of carbon black which are substantially devoid of occluded gases, which includes the steps of mixing carbon black and a non-solvent liquid wetting agent to form a pasty mass, forming the pasty mass into a compressed sheet-like layer by subjecting it to rolling pressures by a pressure-exerting roller member, subdividing said layer to form small aggregates thereof, and thereafter drying the said aggregates at temperatures within the range of approximately 400° to 500° F. while subjecting the aggregates to a rotary tumbling motion, whereby a substantially dry, porous, friable granular product is formed which is substantially dustless.

10. The method of making minute aggregates of dustless carbon black, which includes the steps of mixing dry carbon black and a liquid wetting agent to form a pasty mass, rolling the mass with a pressure-exerting roller member to form a sheet-like layer therefrom, subdividing said layer to form small aggregates of the carbon black and liquid, and thereafter drying said aggregates by removing the liquid wetting agent at temperatures within the range of approximately 400° to 500° F. while subjecting the aggregates to a rotary tumbling motion, whereby substantially dry, porous, friable granular dustless particles of carbon black are produced.

HANS WALTER GROTE.

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Aug. 16, 1938. H. W. PRICE 2,127,137

APPARATUS FOR TREATING FINELY DIVIDED POWDERS

Filed Jan. 17, 1934

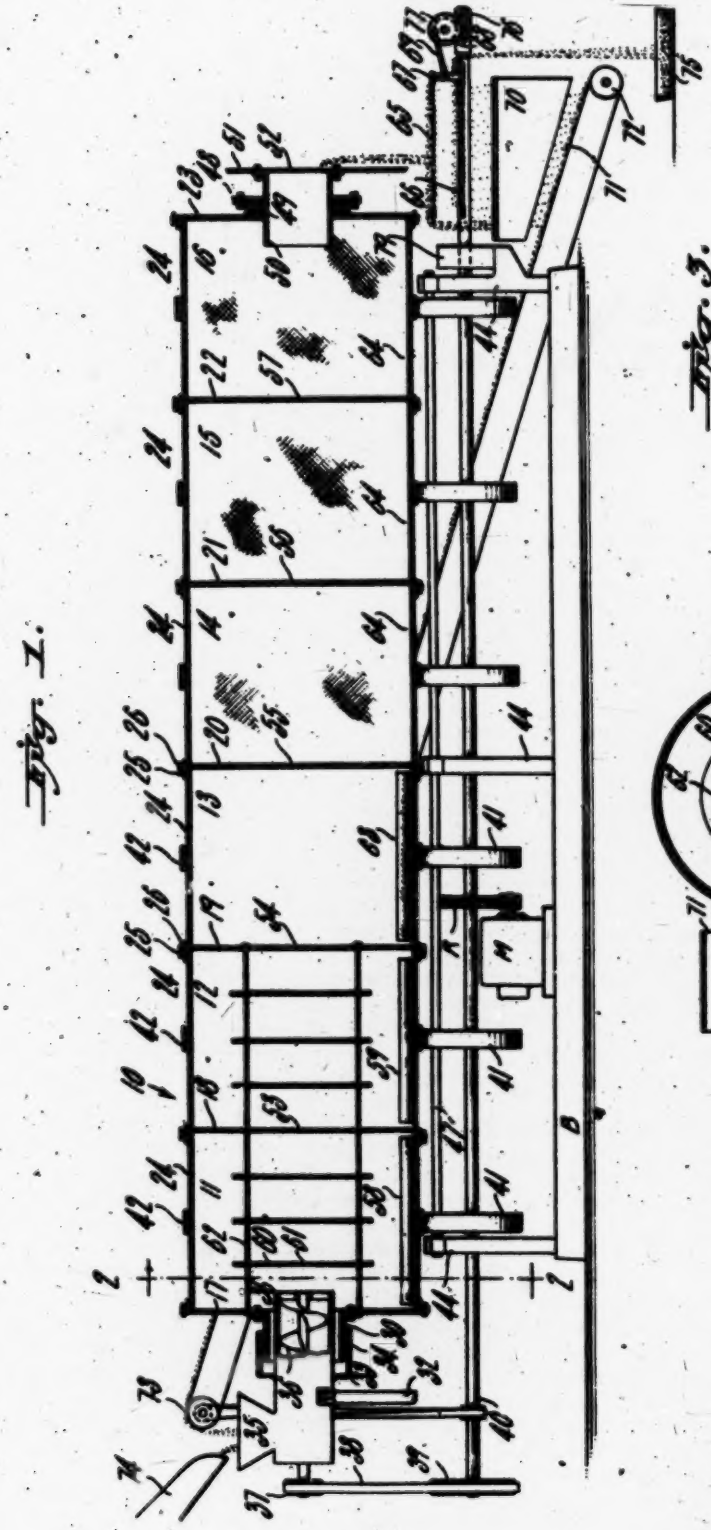


Fig. 1.

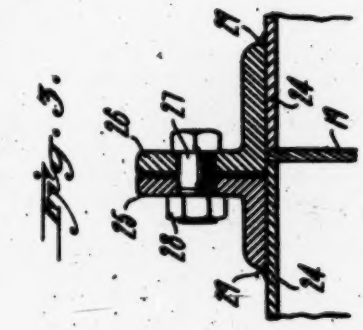


Fig. 3.

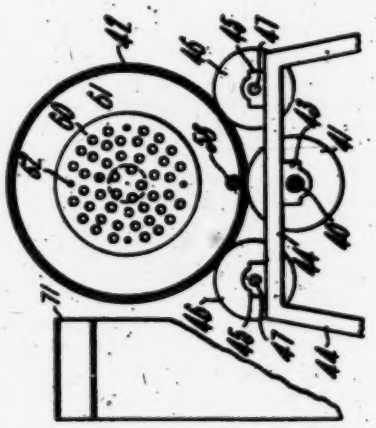


Fig. 2.

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Patented Aug. 16, 1938

2,127,137

UNITED STATES PATENT OFFICE

2,127,137

APPARATUS FOR TREATING FINELY DIVIDED POWDERS

Howard W. Price, Borger, Tex., assignor, by mesne assignments, to J. M. Huber Corporation, Borger, Tex., a corporation of Delaware

Application January 17, 1934, Serial No. 706,909

3 Claims. (Cl. 259—3)

In commercial form there are at present manufactured many fine powders in a dry or practically dry state such as precipitated magnesium carbonate, calcium carbonate, zinc oxide, barium sulphate, talcs, clays, and various forms of carbonaceous blacks known as carbon black or channel gas black, lamp black and other carbons manufactured by the decomposition of carbonaceous gases and used for compounding with various substances such as rubber compounds, paints and varnishes, printing inks, sound recording material and various wax and resinous pyroxylins.

A well recognised difficulty in handling all of these fine powders is that they sift through the containers used to confine them. Moreover, when packed in barrels or paper sacks, an excessive dusting occurs when the products are being used or mixed with other ingredients, and as a result the area surrounding the points or points of handling is filled with dust which settles on surrounding objects, contaminates other products, and pollutes the air which those working with the product have to breathe.

Carbon black is illustrative of this type of material and of the difficulties encountered in making and using it. This product is particularly hard to handle due to the fact that it is a very light and finely divided material composed entirely of amorphous carbon particles and without any binder. As it comes from the decomposition houses and through various bolting and sifting apparatus, it has a weight of only two to four pounds per cubic foot.

Before this black is packed into commercial containers, usually paper bags, it is customarily placed in suitable bins, having within them revolving shafts to which are attached blades or pins. In these bins the black is agitated or stirred to agglomerate the particles and to increase their weight per cubic foot by disassociating from the particles the air and gases which have become entrained in them.

It is not always desirable, however, to carry out this agitation to a point where the black exceeds a weight from twelve to fifteen pounds per cubic foot as further compressing by this means is likely to eliminate some of its characteristics of easy incorporation in certain oils and varnishes such as are used in the paint and ink trades. However, for the purpose of compounding with other more viscous materials such as rubber, where large quantities are used, it has been customary to compress the black further by means of heavy hydraulic or mechanical presses to a state where its weight is from twenty to thirty pounds per cubic foot. This briquetting process is of advantage to prevent sifting and dusting in handling, as well as to reduce the amount of storage space required, although it involves a number of disadvantages as will hereinafter appear.

Practically all modes of handling black require a preliminary bin agitation such as that briefly referred to above. This method of prolonged stirring in order to eliminate entrained air and gases is almost uniformly used as a preliminary procedure for any process in which the carbon black particles are mechanically treated. It has also been found that this treatment has its limitations, for if agitation be carried to a point where the black is reduced to an apparent density of more than fifteen pounds per cubic foot it is necessary to use a relatively large amount of power and to run the risk of fire by reason of the great internal friction set up between the black particles and between the black and the agitation blades or the sides of the bins.

Further to reduce the density of the black to facilitate easier handling, to reduce storage space, and to produce a firmer agglomeration of the particles to minimize dusting, several methods are in use.

One such method is to subject the carbon black to multi-directional pressure by violently agitating and compressing it, thereby causing the particles to be hammered into aggregates of spherical form. This is in reality an extension of the bin agitation principle and although the resultant product may sometimes be found to be satisfactory, the process requires high speed machinery and a large amount of power, thus making it expensive, particularly as only limited quantities of carbon black can be treated at one time in the apparatus necessitated by this procedure. The clearances between the various moving parts of the apparatus used in this treatment, as well as other conditions, require extremely fine adjustment and control which are obtained and maintained with difficulty, and unless these various conditions are properly regulated, the aggregates are likely to become broken up and sheared, with little or no benefit having been derived from the treatment. Moreover, the high speeds necessary properly to agitate the carbon black particles by this method are likely to cause an excessive frictional heat to be developed in the carbon black charge, thus creating the danger of losses by fire.

As previously indicated, it has also been com-

mon practice in the art to utilize various means in order to apply heavy pressures to aggregates or carbon black in order to compress them. This procedure, although it does not overcome the disadvantages of handling the dusty carbon black preparatory to compression, does reduce the volume of the black as well as its tendency subsequently to "dust", and carbon black in this compacted condition is commercially sold, having a density close to twenty-five pounds per cubic foot. Often the black is compressed in bags or other containers which become wrinkled and crimped during the pressure treatment, thus trapping a considerable quantity of carbon black in the wrinkles, and this entrapped black can only be removed by violent shaking and then only at the expense of a cloud of dust. It is obvious that the application of heavy pressures involves heavy and expensive machinery, a large amount of power, increased expense and delay in preparing the product for shipment, while the product is very likely to be caked so hard that it is difficult to disperse or to mix it with other material.

Another mechanical procedure used to render carbon black more dense and capable of being more easily handled is the application of pressure by passing it through the rollers of a Wheatley press. This procedure is carried out after an initial bin agitation which brings the product down to a density of from twelve to fifteen pounds per cubic foot, and involves compressing the black in a screw conveyor and delivering it to and passing it through several pairs of rollers capable of pressing and consolidating it. Such a procedure, although possessing the advantage of continuity of treatment, produces hard flakes which do not readily break down, as on a rubber mill, and which must be forced through a screen to break the flakes apart again, and this usually involves the production of a considerable quantity of the same fine dust which was originally sought to be avoided. Moreover, the amount of black which can be passed through one machine of this type is limited and production is retarded to this extent.

There have been other methods suggested of reducing the apparent density and dustiness of carbon black, notably those involving the addition of tarry binders and by wetting the charge with a suitable agent or agents. These methods, however, are not strictly mechanical in nature and, therefore, will not be discussed in detail.

In my co-pending application Serial No. 703,402 filed Dec. 21, 1933, I have disclosed and claimed a continuous treatment contrasting sharply with previously proposed methods of mechanically treating carbon black in order to retain its desirable characteristics but to eliminate its undesirable ones. The present invention is directed to an apparatus by means of which this novel method may be accomplished, with the elimination of the disadvantages inherent in previously known apparatus, necessitated by the processes which they were required to carry out. The apparatus of the present invention is applicable to the treatment of various kinds and forms of finely divided material although it is described in this specification in connection with the treatment of carbon black for purposes of illustration.

Objects of the invention will be seen to include the provision of an apparatus which circumvents the difficulties heretofore faced in the aggregation of finely divided material such as carbon black, and which is simple, efficient and economical in operation.

A further object is to provide an improved apparatus for making a product which is chemically the same as the original finely divided material, but which has an increased density, and is in the form of granules or pellets which are soft, substantially dustless, easy to handle and capable of being readily dispersed in or intermixed with the material with which it is customarily used, yet which has bulk characteristics which enable it to flow, as through chutes and conveyors, and to be transported in tank cars or tank trucks without material disintegration.

I have discovered that it is possible to change finely divided material such as carbon black to a dense, free-flowing, non-dusting state of agglomeration by mechanical means without placing it in or passing it through presses and without preliminary or violent agitation. This may be done by an apparatus which subjects the material to a continuous tumbling agitation by advancing the black through a predetermined course within a revolving drum or drums in certain of which are included a series of relatively light, free rollers which continue to eliminate the entrained gases and air. After being subjected to this treatment the particles become agglomerated into a product of the desired density. While carbon black may be previously agitated in the regular way before being subjected to treatment, it may also be taken just as it comes from the bolting or sifting apparatus without initially stirring or agitating it in bins, as explained above, where heretofore it was required to be brought to a density of approximately twelve to fifteen pounds per cubic foot before it was ready for further processing. The black is subjected to a progressive moderate pressing, rolling and tumbling of the particles which effects a continuous gentle agitation by virtue of which the black can be processed continuously and brought directly to an apparent density of twenty-five or more pounds per cubic foot. This agitation may be seen to involve a treatment of the particles in an unconfined state, without the use of heavy confining pressure, and the treatment effects the agglomeration of the particles. They are subsequently separated into coherent polyhedral agglomerates of a size that can be handled without dusting. The process is continuous and, therefore, well adapted to quantity production, and separate preliminary or accessory treatments and their accompanying cost, equipment and labor are unnecessary. The product is of a substantially uniform standard of quality and well adapted to "flow" without dusting; it is compact and easily handled without substantial loss by dispersion in air, and is readily dispersible or miscible with the types of ingredients with which it is ordinarily used.

Referring to the drawing, Fig. 1 is a side elevation, partly in section, of the apparatus of the present invention; Fig. 2 is a section taken through line 2—2 of Fig. 1 with the conveyor structure omitted; and Fig. 3 is a detail of a preferred type of connection between the compartments of the revolving drum of Fig. 1.

Briefly described, the apparatus consists of a revolving drum 10 provided with compartments 11, 12, 13, 14, 15 and 16 into the front end 17 of which drum the carbon black particles are delivered for tumbling and agitation. As the particles are tumbled about in the drum, they

pass slowly from one compartment to another and finally emerge from the last drum 16 in a substantially uniform state of agglomeration.

The compartments in drum 10, which is preferably of iron or steel, are formed by circular iron or steel plates 18, 19, 20, 21 and 22, secured between cylindrical members 24 as by brackets 25 and 26. The preferred means for securing these members together, in order to ensure a smooth interior, is indicated in Fig. 3, the brackets 25 and 26 being welded to the cylinders 24 as at 29, and fastened together by turning nut 28 on bolt 27. By welding the brackets in proper position, one of them is made to overlap the adjacent circular plate, such as is indicated at 19, and also to allow the sections 24 to be drawn tightly against the plate to provide a tight and rigid connection.

At the feeding end of the machine, end plate 17 of drum 10 has bolted thereto a flanged cylinder 30 into which is extended the delivery end of a screw conveyor 31. The conveyor housing is supported by suitable supports 32 secured to any suitable framework. To the housing of the screw conveyor is fixed member 33 extending around the flanged cylinder 30 and provided on its inner face with packing material 34 such as graphited fabric or cord in order to seal the joint and prevent the treated material from "dusting" therethrough.

The shaft of the screw 36 of the conveyor may be provided with a pulley 37 driven by belt 38 passing over pulley 39 on shaft 40. On shaft 40 are also provided wheels 41 arranged frictionally to contact against tires 42 secured to the outer surface of each cylinder 24. Shaft 40 is journaled in bearings 43 (see Fig. 2) secured to standards 44 on base B. The shaft 40 is driven by any suitable source of power M applied through reduction gears R.

The drum structure is also carried by standards 44 which carry bearings 45 for holding shafts 47 which in turn carry a number of rollers 48 upon which the drum assembly 10 rests and rotates on being driven through wheels 41 and tires 42. This supporting structure is clearly seen in Fig. 2. If desired, the shafts 46 and wheels 41 may be omitted and one set of rollers 48 arranged to impart rotation to the drum.

The exit end of the drum assembly is closed by plate 23 to which is secured a gland 48 containing packing 49. This packing is pressed against another flanged cylinder 50 secured to a fixed member 51 of the framework of the machine. Member 50 extends into the last compartment 16 of the drum assembly and member 51 is provided with an opening 52 to allow the aggregates of material to pass through it.

It will be seen that the above described construction provides a sealed joint at the exit or discharge end, as well as the feeding end of the apparatus.

In each circular plate 18, 19, 20, 21 and 22 is provided an opening 53, 54, 55, 56 and 57. These openings are of a size sufficient to permit the continuous flow of material from one end of the apparatus to the other, and this occurs as a "head" is built up in the feeding end, due to the flow of material through screw conveyor 31. It will be observed that the partitions serve to limit the longitudinal movement of the rollers within the drum and to localize their action to an extent which will prevent the treated material from flowing too quickly from their zone of action. The partitions may, therefore, be made so that relatively large openings 53, 54, etc. are provided.

In the first two compartments relatively long light rollers 58 and 59 of wood, rubber surfaced metal or other suitable material, are provided so that they roll about the inner surfaces of the cylinder 24 as they rotate. Baffles 60 provided with apertures 61 to prevent the treated material from clogging the apparatus, may be secured in place by rods 62 in turn secured to the plates 17, 18, and 19. These baffles tend to prevent dust, which is present in large quantities in the first two drums, from flying into the other drums. In some cases it may be found desirable to dispense with these baffles 60.

The compartment 13 may be provided with a wire screen roller 63 having suitable openings, for instance, a screen of one-half inch mesh. This roller may, however, be replaced by a solid roller of the type described for compartments 11 and 12.

Compartments 14, 15 and 16 contain no rollers but are lined with wire screen material 64 of appropriate size, one-half inch mesh being satisfactory in the treatment of carbon black. These screen linings may be omitted if desired.

Directly underneath the exit end of the machine there may be provided shaker screens 65 and 66 sliding in suitable guides (not shown) and connected together by link 67. These screens may be agitated by eccentric 68 operating through connecting rod 69. Below the screen 66 is a chute 70 terminating above conveyor 71. Conveyor 71 may be of any desired type but is illustrated as consisting of a belt stretched between drums 72 and 73, the later being positioned over hopper 35 of the screw conveyor. The belt may be driven from any suitable source of power applied to the shafts of drum 72 or 73.

The screens 65 and 66 may be dispensed with by proper regulation of the conditions of the process.

In operation, power is supplied to drive shaft 40 and finely divided material such as carbon black is fed from chute 74 into hopper 35 and conveyed by screw 36 through member 30 and into the first compartment 11 of the apparatus. Shaft 40 drives the screw conveyor through pulleys 39 and 37 and belt 38, and also causes the drum assembly to rotate by reason of wheels 41 rotating against tires 42. The drum assembly is rotated rather slowly, say 10 R. P. M., so that the material is tumbled and thereby agitated and agglomerated in the various compartments and passes slowly from one to the other until the last one is reached.

In giving a specific angular speed of 10 R. P. M., it should be remembered that this is illustrative merely, and is a satisfactory speed of operation when treating carbon black. The speed required in any given case should be adjusted as the conditions of such case require. In the main, the principle to be observed is that the drums or compartments, with rollers removed, should not rotate so fast that the material is carried upwardly and caused to pour back into the center of the treated mass, but should be only fast enough so that it will roll down the inner surface of the drums and thus be subjected to the gentle tumbling action sought to be imparted.

The supply of treated material is preferably controlled so that each compartment is somewhat less than one-half full. Of course, the machine might be operated with the compartments more than half full, or considerably less than

half full, but in order to operate at maximum efficiency consistent with maximum rate of flow, the operation should be carried out as indicated.

In general, the effect of the rollers in the first two or three compartments is to give a gentle rolling pressure by which entrained air and gases may be eliminated and the density of the black increased. The inner surfaces of compartments 14, 15 and 16 give a gentle tumbling action and facilitate the formation of granules. As the drum assembly rotates, the particles are first made more dense and afterwards collect in substantially uniform agglomerates within the desired range of sizes, by reason of the continuous gentle pressing and rolling treatment and tumbling action, to which they have been subjected, and they gradually pass from chamber to chamber until they emerge from cylinder 50 through opening 52 and drop down on chute leading to a conveyor 75 for transmission to the packing point, or upon the shaker screens 65 and 66. At this point the whole mass has become agglomerated into aggregates which are quite compact and which possess considerable uniformity in size. When screening is desired, the screening apparatus is operated through shaft 40 by clutch and gears 79, worm 76 and gear 77. This operates the eccentric 68 and causes the screen to reciprocate. The screens are of predetermined mesh and for carbon black I may state that a very desirable product may be obtained by making the screen 65 of 20 mesh and screen 66 of 65 mesh. Particles smaller than 20 mesh will drop through screen 65 and particles smaller than 65 mesh pass through screen 66 and drop down in chute 70. The particles which fail to drop through screen 65 are undesirable because they are too large and they are removed at short intervals during the operation of the machine and led to conveyor 71. If desired, the screen 65 may be tilted to cause these particles to fall directly upon conveyor 71. Conveyor 71 brings them to the other end of the machine and drops them in hopper 35 where they are again put through the apparatus in order to bring them down to the required size. The particles which come through the screen 66 are too small and, as indicated in Fig. 1, they are dropped to the chute 70 and delivered to the conveyor 71 where they, together with the larger particles, are brought to the feeding end of the apparatus and again put through it in order to build them up into more desired agglomerates. The particles which remain on the screen 66 are of the desired size and in the instance illustrated will be of a size ranging from 20 to 65 mesh. These are collected on the conveyor 75 and brought to a suitable packing and shipping station. Screen 66 may be tilted to facilitate the passage of these agglomerated particles to conveyor 75.

In the case of carbon black, I have discovered that by subjecting the finely divided material to a treatment in the above described apparatus, the particles between 20 and 65 mesh comprise a substantial proportion of the total amount and as these are of a size eminently suitable for the purposes in mind it is apparent that the apparatus is very efficient in achieving its purpose.

In adapting the apparatus for the treatment of a particular material, it should be observed that the number of drums or compartments and the angular speed thereof are factors determinative of the uniformity of the size of the granular end product and, therefore, should be considered in determining the necessity for screens for selective separation. The weight of the rollers in the first two or more drums is, on the other hand, determinative of the density of the product, and by proper choice of rollers the desired number of pounds per cubic foot of end product may be obtained.

It may be pointed out also that instrumentalities shown in the drawing and referred to above in this specification are illustrative merely of a preferred embodiment of the invention. It will be understood, however, that the baffles 60 in compartments 11 and 12 of the device, while desirable, may be eliminated. Any form of conveyor may, of course, be used instead of those shown at 71 and 75.

What I claim is:

1. An agglomerating apparatus comprising in combination a rotatable drum divided into cylindrical compartments longitudinally thereof by partitions, means for feeding finely divided solid material into the first compartment, central openings in the partitions whereby the material supplied to the first compartment is allowed to overflow into the next and successive compartments from one end of the drum to the other upon rotation thereof, and a light free roller within each of a predetermined number of the first of said compartments also operable upon rotation of said drum, the subsequent compartments being free of rollers.

2. An agglomerating apparatus comprising in combination a rotatable drum divided into cylindrical compartments longitudinally thereof by partitions, means for feeding finely divided solid material into the first compartment, central openings in the partitions whereby the material supplied to the first compartment is allowed to overflow into the next and successive compartments from one end of the drum to the other upon rotation thereof, and a light free roller within each of a predetermined number of the first of said compartments also operable upon rotation of said drum, the subsequent compartments being free of rollers, and a discharge opening in the end wall of the last compartment.

3. An apparatus for agglomerating carbon black comprising a rotatable drum, a plurality of members disposed within the drum transversely thereof forming partitions therein dividing the drum into sections, a plurality of light free rollers each disposed within a separate predetermined section at one end of said drum sections at the other end of the drum being free from rollers, said partitions having central perforations whereby said black supplied to the first section may flow from end to end of the drum with a tumbling action upon rotation of the drum.

HOWARD W. PRICE.

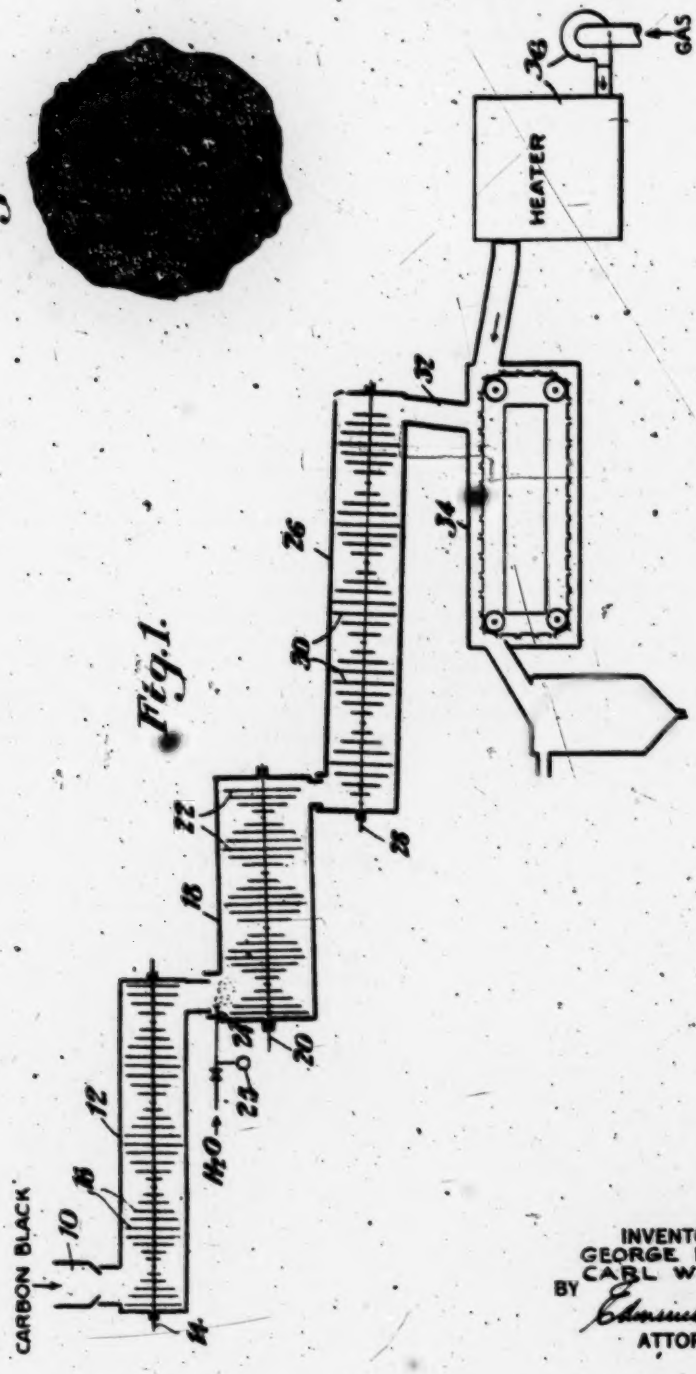
Sept. 27, 1938. G. L. HELLER ET AL 2,131,686

GRANULAR CARBON BLACK AND METHOD FOR MANUFACTURE

Filed May 9, 1936

Fig. 2.

Fig. 1.



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Patented Sept. 27, 1938 2,131,686

UNITED STATES PATENT OFFICE

2,131,686

GRANULAR CARBON BLACK AND METHOD FOR MANUFACTURE

George L. Heller and Carl W. Snow, Pampa, Tex., assignors to General Atlas Carbon Company, Dover, Del., a corporation of Delaware

Application May 9, 1936, Serial No. 78,836

5 Claims. (Cl. 134—60)

This invention relates to the agglomeration or pebbling of carbon black, and particularly concerns a new form of granular black and process of making the same.

Carbon black as originally produced by dissociation of hydrocarbons, and as commonly employed in the art of rubber compounding, is a finely divided impalpable powder which is extremely difficult to handle because of its light bulk and dusty character. As originally produced carbon black usually has an apparent density of 3 to 5 pounds per cubic foot and an apparent individual particle diameter in the range of .05 to 1 micron. It is the small size of these individual particles which imparts to the black its colloidal properties of uniform dispersibility in rubber, oils, resins, etc. Even these apparent individual particles, however, are in reality themselves primary agglomerates of smaller particles which are invisible to the most powerful microscope.

The primary object of the present invention is to provide an improved process for efficiently and economically agglomerating carbon black to a granular condition in which it is free-flowing, non-dusting, sufficiently strong to withstand handling, and retains its original property of uniform dispersibility when milled with rubber, oil, or the like.

It has long been known that carbon black can be agglomerated to a granular condition through the mixing and wetting of the particles of black with a suitable liquid such as gasoline or kerosene, and their concretion into grains. A feature of the present invention resides in the discovery that under properly controlled conditions of wetting and agitation, carbon black can be agglomerated by means of a single phase liquid such as water to produce a free-flowing, non-dusting granular product which is readily and uniformly dispersible when milled with rubber or oil, and which is stronger and denser than similar granular black prepared by using other wetting agents such as kerosene, gasoline, etc. Another feature which is unique is that this process is applicable to all types of carbon black, including the "soft blacks" and lampblack in an economical and efficient manner. It has been found that this process will convert above 90% of the dry powder to agglomerates ranging between 40 mesh and 10 mesh on screening.

With the above and other objects and features in view, the invention consists in the improved granular black and method for agglomerating carbon black to produce this product, as hereinafter described and more particularly defined by the accompanying claims.

In the following description reference will be made to the accompanying drawing, in which Fig. 1 is a diagrammatic flow diagram of apparatus adapted for practicing the invention; and Fig. 2 is a sectional view of a single grain of the granular product, magnified.

Referring to the accompanying flow diagram, the finely divided black which is to be agglomerated is continuously charged from a hopper 10 into a horizontal tubular conveyor 12 of the screw conveyor type. Instead of the usual flights comprising a screw conveyor, conveyor 12 is shown equipped with an axially mounted rotating shaft 14 carrying radial rods 16 having a length about 1" less than the internal diameter of the conveyor, disposed at uniform longitudinally and angularly spaced distances throughout its length. It has been found that a conveyor of this design having a length about six times its diameter and having a central shaft with radial rods set about 1" apart and spaced 45° radially, rotated at about 30 R. P. M., will increase the density of black about 100% while conveying it under unidirectional rotation of the rods, from one end of the conveyor to the other. This densing effect is apparently the result of a number of factors including (a) agitation of the black while mechanically conveying it through the conveyor 12 which is completely filled with black being forced therethrough by the rotation of the conveying and agitating mechanism, and (b) the agglomerating and impacting action of the rods 16 to which the particles of black adhere and then build up by impact into larger particles which are less adherent and therefore drop off the rods, as the rods are forcibly drawn through the body of dry particles. The amount of increase in apparent density of the black which is effected by this operation may be controlled by varying the rate of rotation of shaft 14. The more the black is pre-densed, the greater the efficiency of the subsequent wetting and agglomerating operations.

After pre-densing the black to the desired degree (preferably to at least 12–15 pounds cu. ft.) in unit 12, the black is continuously discharged into the feed end of a mixer 18. Mixer 18 is a cylindrical tube or trough which may be mounted horizontally, but is preferably mounted with its major axis tilted from the horizontal so that the discharge end is a few inches higher than the feed end. The agitator element in the mixer preferably comprises an axial rotating shaft 20 on which radial rods 22 are mounted at uniformly spaced distances both longitudinally and angularly throughout the length of the shaft. One such mixer which worked satisfactorily consisted of a cylinder 20" in diameter and 60" long having an inner enamel lining and equipped with an axial rotating shaft 20 with rods 22 of ⅜" diameter and 19¾" length mounted radially on the shaft to extend diametrically of the cylinder at 1½" longitudinal spacing and 22½° radial spacing between adjacent rods. Each end of a

rod 22 functions as an agitating finger rotating within the cylinder at the rate of 180 R. P. M. In moving through a unit inch and a half length of path beginning in the plane of rotation of one rod and ending in the plane of rotation of the next adjacent rod, a unit volume of the charge of black in the mixer is impacted by both ends of the rod at one end of the inch and a half path and also by both ends of the rod at the other end of the inch and a half path. In other words the charge in each thus measured unit inch and a half length of the mixer is impacted at the rate of twelve impacts per second, or at the rate of approximately eight impacts per second per linear inch of path. However, in moving through an inch and a half length of path having its center point located directly in the vertical plane of rotation of the two ends of one rod, the black would only be impacted by those two fingers and therefore would receive impacts at the rate of six per second per linear inch and a half of path, or at the rate of approximately four per second per linear inch of path. The charge advances through the mixer very slowly at a rate far less than a linear inch per second. It will thus be apparent that the charge in the mixer is subjected to impacts by the rotating fingers at an average rate of four to eight per second per linear inch of path followed by the charge through the mixer.

The dry preferably pre-densed black which enters the mixer is sprayed with water supplied at air temperature from one or more sprays 24, as the black is fed into the feed end of mixer 18. By means of valves and a meter 25 the amount of water is accurately proportioned to the rate of supply of dry black to the mixer so that the water and black are usually mixed in the proportion of approximately 48% water–52% black by weight. The allowable proportions of liquid and black vary for the material and liquid but lie within the range from 29% to 65% liquid by weight of finished wet product. For example, using water for the liquid, commercial rubber channel black may require as much as 56% water, while the "soft blacks" may require as little as 43% water by weight of product. By rotating the mixer shaft at a speed of about 180 R. P. M. it has been found possible to partially wet the black and agglomerate the wetted particles into small nucleal granules during its passage from the feed to the discharge end of mixer 18. By rotating the mixer shaft at this speed the black and water are both kept in a state of finely divided suspension during the wetting operation. The tilted position of the mixer helps to hold the water and dry black in the lower feed end, while the agglomerate nuclei are selectively conveyed by the uni-directional rotation of rods or fingers 22 toward the higher discharge end of the mixer.

From the discharge end of mixer 18 the moist nucleal granules are delivered to the feed end of an agglomerating cylindrical agitator 26 which is preferably lined with enamel. It has been found that the agitator 26 should be proportioned relative to the mixer so that it is of about the same or greater length, and half the diameter, of the mixer. Also it is advantageous though not necessary, to have the agitator slightly tilted so that its discharge end is a few inches (2"–4") lower than the inlet end in order to permit of a certain amount of gravity classification of the granules as they are conveyed therethrough. The agitating element of member 26 preferably comprises an axial shaft 28 with radial rods 30 mounted thereon at uniform longitudinal and radial spacings, and rotated at about one-half the rate of speed of the agitator element of mixer 18. A satisfactory assemblage for member 26 was found to be a trough having a cylindrical bottom of 6" radius and 60" long having rods ⅜" in diameter, 11⅝" long spaced 1½" longitudinally and 30° radially on a shaft 28 rotated at a speed of 90 R. P. M. During passage through agitator 26 the small moist nucleal granules are built up and agglomerated into fairly uniformly sized dense grains of rounded contour and uniformly porous but relatively strong texture. These grains are composed of a large number of nuclei and possess no particularly uniform shape but may be made essentially spherical by prolonged agitation in member 26.

From the discharge end of agglomerator 26 the moist granular product is conducted by a chute 32 into a drier 34 wherein the black granules are subjected to rapid (flash) drying by means of a current of high temperature gas or air blown thereover from a blower-heater unit 36. The temperature to which the granules are subjected in drier 34 may advantageously be as high as 400–600° F. in order to rapidly and completely dry the granules. The dried granular product has an apparent density in the neighborhood of 28–40 pounds per cubic ft. Approximately 90% of this granular product will pass through a standard 10 mesh screen but only about 2% will pass through a 40 mesh screen. Only about half of the product passing through the 10 mesh will pass through a 16 mesh screen. A yield has been obtained as high as 95% of product passing through a 10 mesh and retained by a 40 mesh screen.

Carbon black is wetted much more readily by many liquids than by water, as for example concentrated acetic acid, alcohols, benzene, or acetone. Nevertheless the efficiency of any particular liquid in effecting agglomeration of carbon black is not entirely the result of its ability or rapidity in wetting the black but rather the combination of ability to wet and yet wet slow enough so that the liquid may be dispersed in small droplets before the formation of the nuclei is completed. Thus benzene wets so rapidly that it cannot be used, as do all liquids having a specific inductive capacity less than 10 (see chemical handbook for values).

If care is taken to insure uniform wetting by sufficient agitation and if the oil content of the carbon is not too high (should be below 0.2%), water is much more efficient than most other liquids as an agent for promoting granulation of black because it combines the characteristics of relatively high surface tension (73 dynes per cm² at 20° C.) and not too low a dielectric (81), which seems necessary in order to insure that the black be wetted without too much difficulty while at the same time producing a product having a higher uniform density and stronger texture than would be obtainable using a liquid such as acetone, with surface tension 23 and dielectric 26.6. Some liquids of lower dielectric are more efficient wetting agents but are nevertheless not as efficient as agglomerating agents. By blending two miscible liquids such as water and acetone, the wetting efficiency can be increased at the sacrifice of a proportionate reduction in the strength and density of the finished granular product. It has been found that liquids possessing a specific inductive capacity (dielectric) less than 10 (based on wave lengths above 10,000 cm.

are not suitable for a wetting agent and cause the black to form a pasty mass which will not form granules. For normal operation water is the most suitable liquid.

In the mixer 18 the dry black is partially wetted with about an equal weight of water, the action of the rotating fingers or bars 22 being to drag drops of water through the black, thereby gradually wetting the particles and coalescing and compacting them with the aid of surface tension and dielectric effects into small spherical agglomerate nuclei of such small size as to readily pass a 100 mesh screen. Further agitation and compacting in the agitator 30 agglomerates these nuclei into grains of somewhat rough but porous surface, varying in size between approximately 8 and 40 mesh. While the grains are non-dusting, their outer surface is rough and any unagglomerated black readily adheres thereto. When grains of the finished and dried granular product are broken and viewed under a microscope, they will be found to consist of an irregular pattern of compacted nuclei which is uniform throughout both as to porosity and density, as shown by Fig. 2.

Rubber tests comparing treated black and untreated black show that the granular form disperses with about the same rise in internal temperature as the original black and that the power requirement to mill the granular form into a compound is about 15% lower.

The invention having thus been described, what is claimed as new is:

1. The continuous process of treating flocculent carbon black to p. duce a free-flowing nondusting granular product, which comprises increasing the apparent density of the black to about 12 pounds per cu. ft. by feeding it under mechanical stirring and pressure through a horizontally disposed tubular conveyer, thereafter wetting the dry pre-densed black with approximately 48% by weight of the mixture of water and agitating the wetted mixture to form agglomerate granular nuclei while selectively removing said nuclei from the wetting zone, further agglomerating the nuclei by agitation and gravity classification to produce free-flowing substantially uniform granules, and drying said granules by moving them rapidly through a drying gas atmosphere maintained at a temperature of approximately 400° F.–600° F.

2. The continuous process of treating flocculent carbon black to produce a free-flowing nondusting granular product, which comprises increasing the apparent density of the black to at least 12 pounds per cu. ft. by feeding it under pressure through a tubular conveyer, thereafter agitating the black and water in the proportions of 47–49% water to 53–51% black under controlled conditions of agitation to effect wetting, formation of granular nuclei, and selective removal of said nuclei from the wetting zone, further agglomerating the nuclei by agitation and gravity classification to produce free-flowing substantially spherical granules, and drying said granules.

3. The continuous process of treating flocculent carbon black to produce a free flowing nondusting granular product of greatly increased density which comprises, feeding the black under mechanical agitation through a horizontally disposed confined path while gradually wetting the dry black with approximately 35%–65% by weight of the mixture of water to form agglomerate granular nuclei, the agitation and advancing of the mixture through the wetting zone being effected by subjecting the mixture to impacts at an average rate of four to eight per second per linear inch of path, imparting said impacts by a plurality of metallic fingers mounted along the path in closely spaced linear and angular relation and moving at high speed in a direction transversely of the direction of advancing movement of the mixture, the speed of movement and spacing of the fingers being such as to keep the black and water in a state of finely divided suspension during the wetting and granular nuclei forming operation, and further agglomerating the nuclei by agitation at a rate about half that employed in the wetting zone to produce free flowing granules of approximately 8–40 mesh size, and drying said granules.

4. The continuous process of treating flocculent carbon black to produce a free flowing nondusting granular product which comprises, feeding the black under mechanical agitation through a horizontally disposed confined path having a length to perimeter ratio of about 1–1 while gradually wetting the dry black with finely atomized water in the proportions of 35%–65% water by weight of black to form agglomerate granular nuclei, agitating and advancing the mixture through the wetting zone by subjecting it to impacts at an average rate of four to eight per second per linear inch of path, imparting said impacts by a plurality of metallic fingers mounted along the path with a linear spacing of about one and one-half inches and an angular spacing of about twenty-two and one-half degrees (22½°), said fingers rotating at high speed in a transverse direction to the direction of advancing movement of the mixture, the speed of movement and spacing of the fingers being such as to keep the black and water in a state of finely divided suspension during the wetting and granular nuclei forming operation, further agglomerating the nuclei while advancing them through a path extension by agitation at a rate about half that employed in the wetting zone to produce free flowing substantially spherical granules of approximately 8–40 mesh size, and drying said granules.

5. The continuous process of treating flocculent carbon black to produce a free flowing nondusting granular product which comprises, increasing the apparent density of the black to about twelve pounds per cubic foot by feeding it under mechanical stirring and pressure through a tubular conveyor, thereafter agitating the black with a single phase liquid having a specific inductive capacity greater than 10 and a surface tension preferably above 20 in the proportions of 35%–65% of liquid by weight of black, effecting the agitation and advancing of the mixture through the wetting zone by subjecting the mixture to impacts at an average rate of four to eight per second per linear inch of path, the speed of movement and spacing of the impacting elements being such as to keep the black and liquid in a state of finely divided suspension during the wetting operation thereby forming granular nuclei, removing said nuclei from the wetting zone, further agglomerating the nuclei by agitation at a rate not substantially more than half that employed in the wetting zone to produce free flowing substantially spherical granules, and drying said granules.

CARL W. SNOW.

GEORGE L. HELLER.



[fol. 300] Excerpt from an Outline of Carbon Black

by I. Drogin, Ph.D.

(Published in "The India Rubber Journal." Sept. 7, 1935, pp. 18-19.)

The latest development in rubber grade carbon black is the dustless form. When carbon black first made its appearance in the Rubber Industry it was in the uncompressed form, generally bulking from 10-13 pounds per cubic foot. It was quite inconvenient to mix this form of black on the open mill. Internal mixers had not yet been developed. The next improvement was fully to compress the black to a bulk ranging from 22-24 pounds per cubic foot. This alleviated conditions, but did not fully correct the trouble, because even compressed black is still dusty to handle.

Carbon black can now be converted to a dustless type with bulk ranging from 22 to 26 pounds per cubic foot. This may be accomplished in several ways after the black is brought into the packing house. The net result is a granular carbon black particle of either spherical or irregular shape. The latter is obtained by agitating a rubber grade carbon black, by a continuous process, in a specially designed apparatus. There is no adulterant added to the black, neither is excessive heat generated during the agitation. The continuity of the process assures a uniform blending of the product. This granulized form of black is quite dustless, and its use is growing in popularity in the Rubber Industry. It is quite a help in reducing the dusting nuisance [fol. 301] from the unloading point to where it is mixed in the Banbury. It is true that when used on the open mill the dustless black, after the first pass, will revert to dusting, but one must not overlook the advantages of cleanliness gained in handling the black up to the mill. Dustless types of black can be moved in tanuk cars, as well as in bag shipments. Dustless types of black mix and disperse readily in rubber, and generally produce as high reinforcement in the rubber stock as the fully compressed black.

[fol. 302] Patents to Which the Court Referred in Its Findings of Fact

U. S. Patents Nos.:

Re 21,379	(Part of	Plaintiff's	Exhibit	1)
1,263,082	("	" Defendant's	"	22)
1,405,439	("	" "	"	22)
1,431,079	("	" "	"	22)
1,286,024	("	" "	"	22)
1,561,971	("	" "	"	23)
1,245,700	("	" "	"	22)
Brit. Pat. 183,430	("	" "	"	22)

Portions of Defendant's Exhibit No. 22

UNITED STATES PATENT OFFICE.

GEORGE C. LEWIS, OF NEW YORK, N. Y.

METHOD OF TREATING FINELY-SUBDIVIDED MATERIAL.

1,263,082. Specification of Letters Patent. Patented Apr. 16, 1918.

No Drawing. Application filed January 25, 1917. Serial No. 144,337.

To all whom it may concern:

Be it known that I, GEORGE C. LEWIS, a subject of the King of England, and a resident of the city of New York, New Dorp, Staten Island, borough of Richmond, Richmond county, and State of New York, have invented certain new and useful Improvements in Methods of Treating Finely-Subdivided Material, of which the following is a specification.

My invention is a new and improved method of treating light flocculent or powdered material, or any substance in a fine state of subdivision so as to better adapt it for handling, shipment or storage, reduce the labor cost involved, and lessen the dusty character of the material.

By means of my invention, I am able to reduce a given mass of such material to a fraction of its former volume without mechanical pressure and without impairing its softness, working qualities or state of subdivision. As a result of the use of my improved process, I am able to reduce the size of the minute interstices or interstitial spaces between adjacent particles, flakes, crystals, or other units of the mass and bring said units of the mass closer together without altering their individual size or character, at least to any material or appreciable extent. These minute interstices are ordinarily filled with occluded gas or atmospheric air which gives to the material its distended or inflated condition and which cannot be satisfactorily removed, to reduce the size of said spaces by said mechanical operations as compression of the mass, or the subjecting of it to a partial vacuum.

In carrying out my process, I first displace the occluded atmospheric air or other gas by a liquid which will not act as a solvent for the mass being treated and will not act to chemically or mechanically change the particles themselves. This treatment may be brought about by thoroughly intermixing the liquid and material in any suitable manner and by means of any suitable mixing machine or by adding either the liquid or the solid to the other. The relative proportions of liquid and solid are preferably such as to form a paste or wet plastic mass although a larger quantity or proportion of liquid may be used if desired without detriment to the process. During the intermixing the liquid forces out and displaces the gas and by surface attraction, capillary attraction or other action, the particles of the mass are brought very much closer together or rearranged and shifted in position so that the interstitial spaces are of very much smaller magnitude when filled with such a liquid than when filled with gas.

After the gas has been expelled by the action of the liquid, I remove the liquid but without any material increase in the size of the interstitial spaces, and in some cases with a further drawing together of the particles and a further reduction of the volume of the total mass. This removal of the liquid may be accomplished by evaporation alone or accompanied by heating at a temperature below that which will in any way adversely or deleteriously affect the material by melting, decomposing, charring, or otherwise. The removal of some of the liquid and particularly at the beginning of the operation, may be secured or facilitated by the treatment of the mass to centrifugal action in a suitable centrifugal drier, strainer or separator. The liquid removing operation may be and preferably is continued until the mass regains its original dryness and is thus ready for shipment, storage or commercial purposes. The liquid removed by evaporation, or otherwise, may be collected or condensed and may be used over again in treating another mass of material.

I do not wish to be limited to any particular kind of apparatus and illustration appears to be unnecessary. The intermixing may be accomplished in any suitable mixing machine and the paste or semi-plastic mass may be carried by a conveyer or other mechanism to the drying chambers, or it may be forced through a flat nozzle into the form of ribbons arranged in rows on a drying tray. These are merely suggestions as to the apparatus which might be employed.

Due to the reduction in the volume resulting from the use of my improved process, a given mass of the material may be stored in a smaller space, shipped in a smaller and lighter container and handled more economically, particularly as to labor cost and as to the losses in the form of rising dust during the handling. The process may be used for treating a large number of different substances and I do not wish to be limited to any particular one.

Merely as an example of a material in connection with which my process may be used to great advantage, I may mention carbon black or lamp black. As these are practically insoluble, there is a great range of readily volatile liquids which may be used to displace the occluded gas and which will not affect the constituent particles of the mass. Preferably, a liquid is employed which, upon evaporation, leaves no sediment or residue, or at least no deleterious or objectionable residue. Distilled water, alcohol, ether, gasolene, or various other liquids are suitable, although if great purity of the product is not absolutely necessary, commercially pure water may be used instead of distilled or chemically pure water.

As a further important feature of my invention, I may utilize my process for not only reducing the amount of occluded gas, but at the same time to thoroughly intermix with the material other substances which may aid in reducing the dusty character and which are not inimical to, but useful in connection with the material in some product into which the material is to be finally incorporated.

For instance, if carbon black or lamp black is to be used in the manufacture of rubber, printing ink, or certain other products, suitable hydrocarbons, colloidal or oleaginous materials may be intermixed with the carbon black or lamp black at the same time the liquid is being intermixed to displace the occluded gas. In the manufacture of printing ink, resin oil is a useful ingredient and in some rubber formulæ, certain waxes, oils, etc., are used. In the latter instance, particularly, only small amounts of such hydrocarbons are permissible and difficulty is sometime encountered in getting them thoroughly intermixed and uniformly distributed in the carbon black.

In carrying out my improved process, I make an emulsion of these ingredients with a liquid if they are insoluble in that liquid or dissolve them in the liquid if they are soluble. A paste may be made using from four to five times the weight of liquid to the weight of powder and as the hydrocarbons or other substances are thoroughly intermixed with the liquid, they will thus become thoroughly intermixed with the powder. It may be then thoroughly dried to evaporate the liquid and the resulting mass will have the hydrocarbon or other material thoroughly incorporated in it and such material will aid in reducing, if not entirely destroying the dusty character of the powder and thus reduce, if not eliminate, the losses incurred by powder carried away by air currents during manufacturing processes. This effects not only a saving in the material but renders the air more healthful and gives greater ease of manipulation of the product as well as the lessened bulk.

If, in carrying out my improved process, it is desired to incorporate waxes, or gum resins, in magnesia or carbon black, I find that gasolene is a very satisfactory liquid as it is a solvent of the ordinary waxes and gum resins but does not affect the magnesia or carbon black.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. The process of treating flocculent or other finely subdivided material to reduce the volume of a given mass to facilitate handling, shipment or storage, which includes displacing the occluded gas in the interstitial spaces with a readily vaporizable liquid and then removing the liquid by evaporation.

2. A process of treating flocculent or other finely subdivided material to facilitate handling, shipment or storage, which includes displacing the occluded gas in interstitial spaces by mixing the material with a liquid which will not dissolve or chemically affect the material and which will form a paste therewith, and evaporating the liquid from said paste.

3. The process which includes forming an emulsion or solution of a small quantity of oily substance and a completely vaporizable liquid, forming a paste of said mixture, and a flocculent or other finely subdivided material, and then evaporating a liquid therefrom.

4. The process of preparing a composition including carbon black or lamp black for commercial use in printing ink, rubber, etc., which consists in making a solution or emulsion of an oleaginous substance in a liquid, mixing said liquid with the carbon black or lamp black and completely evaporating the liquid.

Signed at New York, in the county of New York, and State of New York, this 24th day of January, A. D. 1917.

GEORGE C. LEWIS.

Part of Defendant's Exhibit 22.

UNITED STATES PATENT OFFICE.

CHESTER J. RANDALL, OF NAUGATUCK, CONNECTICUT, ASSIGNOR TO THE GOODYEAR'S METALLIC RUBBER SHOE COMPANY, A CORPORATION OF CONNECTICUT.

PREPARATION OF AND THE MIXING OF PIGMENTS WITH RUBBER AND THE LIKE.

1,405,439. Specification of Letters Patent. Patented Feb. 7, 1922.

No Drawing. Application filed March 24, 1917. Serial No. 157,128.

To all whom it may concern:

Be it known that I, CHESTER J. RANDALL, a citizen of the United States, residing at Naugatuck, county of New Haven, and State of Connecticut, have invented certain new and useful Improvements in the Preparation of and the Mixing of Pigments with Rubber and the like, of which the following is a full, clear, and exact description.

This invention relates to the preparation of and the mixing of pigments with rubber and the like, and has for an object to prevent dispersion of pigment into the atmosphere during the mixing operation, to shorten the mixing period, and to provide a pigment mixture which will produce desired characteristics in the rubber product without impairing the quality of the product.

A pigment is usually mixed with rubber being worked by the rolls of a mill, and is usually added to the rubber in the form of a powder. In this form it exhibits particles isolated from each other by air which tends to permit the particles to easily be dispersed into the atmosphere and discolor different rubber stock which may be undergoing treatment nearby. Further, the quantity of pigment required to impart desired characteristics of color, quality, etc., to the rubber, is of so great mass or bulk in powder form as to cause it to mix very slowly with the rubber. Thus an abnormally long milling period is required to produce a homogeneous product. The addition of an adhesive substance to the pigment during the mixing operation has been tried, for the purpose of binding together the particles of the pigment so that they cannot fly from the mill rolls, and also to reduce the bulk of the pigment and thereby shorten the mixing period. But this expedient while partly accomplishing the purpose in view, cannot always be resorted to with impunity, for the quantity of binder usually required is so great as to materially impair the quality of and in some instances even to spoil the rubber product.

In order to remedy the above disadvantages, the present invention consists, in part, of changing the physical state of a pigment from a loose easily dispersable powder to a compact mass, from which dispersion of particles into the atmosphere during the mixing operation cannot occur. This new product has about one-quarter the mass or bulk of the pigment in its original powder condition, and the small bulk of the pigment permits of it being quickly assimilated by the rubber with consequent shortening of the mixing period. Further, in this new product, the ratio of the binder to the pigment is such that subsequent incorporation of the mixture into the rubber will produce desired characteristics of color, quality, etc., without sacrificing other desired characteristics in the rubber.

One embodiment of the invention will be described in connection with the compounding of rubber with black pigment, such as lamp black. Liquid tar has been sometimes added to lamp black on the mill rolls to bind together and prevent the particles of pigment from dispersion into the atmosphere. However, the smallest proportion of tar and lamp black that can be mixed in this way is about two parts of tar to one part of lamp black. This quantity of tar is far in excess of that actually required to bind the particles together and is in fact so large as to materially impair the quality of the rubber product. Further, greater difficulty is experienced in getting such a mass of tar to uniformly permeate the rubber throughout. To overcome these disadvantages, I initially produce a homogeneous mixture of tar and lamp black, in the proportion of about one part of tar to about four or more parts of lamp black, and subsequently incorporate this product with the rubber.

The process in its preferred form is as follows:

A weighed quantity of lamp black is put into a mixing and kneading machine of the usual and well known type, with about one-quarter of its weight in liquid tar. I then add enough naphtha or similar solvent for tar to moisten the whole mass. The mixing machine is then started and during the mixing operation the dissolved tar is evenly distributed through the mass of lamp black. After the mixing machine is run for a sufficient period to accomplish a thorough saturation of the lamp black by the dissolved tar, steam is admitted to the steam jacket of the mixing machine, and the naphtha is distilled off and recovered in a condenser for further use.

The product of the above described preliminary treatment is in the form of a granular mixture, consisting of small pellets

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ranging in size from material in powdered form to lumps the size of a walnut or larger, and which occupies about one-quarter of the space the lamp black alone originally did in the form of a dry powder. The small particles of the product are stuck together sufficiently to prevent their dispersion under ordinary agitation but are capable of being readily broken up and quickly worked into the rubber during the mixing of the same on the rolls of the mill. These rolls may be heated in the usual way.

Although I have described a specific embodiment of the invention I do not intend to limit the scope of the invention thereto, and I desire it to be understood that wherever lamp black is mentioned in the specification and claims, the words carbon black may be substituted, and that whatever applies to lamp black in this disclosure applies also to the use of carbon black as an equivalent.

Having thus described my invention, what I claim and desire to protect by Letters Patent is:

1. The process of compounding rubber, comprising mixing with the same a mass of pigment in which the particles are bound together by a binder disseminated uniformly throughout the mass of the pigment.

2. The process of compounding rubber, comprising mixing with the rubber, lamp black in the form of a mixture in which the particles are bound together by tar substantially uniformly disseminated throughout the mass of lamp black.

3. The process consisting of forming a fluid mass of tar, a solvent therefor and a pigment, changing the mass to a substantially granular mixture by removal of the solvent, and incorporating the product in rubber.

4. The process consisting of forming a fluid mass of pigment, a binder for the pigment and a solvent for the binder, heating the mixture to evaporate the solvent, and incorporating the product in rubber.

5. The method of compounding a plastic substance with an impalpable powder which consists in causing a concretion of the powder into dry, granular or lumpy form, and then mixing it with the plastic substance.

6. The method of compounding rubber with a substance normally existing as an impalpable powder which consists in wetting the powder and drying it to produce a friable, granular or lumpy condition, and incorporating the dry, granular substance with rubber.

7. The method of compounding rubber with lampblack which consists in wetting the powdered lampblack to concrete it, and drying the same, bringing it to a condition of friable grains or lumps, and incorporating the dry, granular lampblack with the rubber on a heated mixing mill.

8. The method of compounding a plastic substance with an impalpable powder which consists in mixing the powder with a concreting and binding liquid, drying the same, producing a condition of friable grains or lumps, and mixing it with the plastic substance.

Signed at Naugatuck, Conn., this 19th day of March, 1917.

CHESTER J. RANDALL.

Patented Oct. 3, 1922. 1,431,079

UNITED STATES PATENT OFFICE.

GEORGE WILSON ACHESON, OF NEWARK, NEW JERSEY, ASSIGNOR TO ACHESON CORPORATION, OF NEW YORK, N. Y., A CORPORATION OF DELAWARE.

METHOD OF PREPARING PIGMENT OIL COMPOSITION.

No Drawing. Application filed August 7, 1922. Serial No. 580,281.

To all whom it may concern:

Be it known that I, GEORGE WILSON ACHESON, a citizen of the United States, residing at Newark, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Methods of Preparing Pigment Oil Compositions, of which the following is a specification.

This invention is a novel method applicable in the preparation of pigment suspensions, including paints, inks, varnishes and analogous products comprising a pigment and a vehicle. The pigments employed in accordance with the present invention are preferably subjected to a preparatory treatment by the procedure known as "deflocculation," such procedure involving, as is now well understood by those skilled in this art, the mechanical working of the pigment material in paste form in conjunction with certain organic bodies, such as tannin, extracts from roasted cereals or other starchy materials, etc., known as deflocculating agents. My invention is not however limited to the employment of pigments which have been subjected to deflocculation as above described, but may be practiced, although less advantageously, with any suitable pigment material in a sufficiently fine state of subdivision.

A typical procedure for the preparation of such pigment suspensions in accordance with my invention is as follows, it being however clearly understood that the invention is not restricted to the particular materials, proportions or manipulations set forth by way of example and illustration:

A suitable pigment such for example as ultramarine, zinc white, carbon black or the like is first, preferably, subjected to deflocculation in known manner, whereby it is wholly or partially converted into a colloidal or permanently suspensible condition. The resulting paste is thinned with water to the consistence of a thin cream. In case of pigments existing in a state of sufficient fineness the deflocculation step may be omitted, but the product in such case is distinctly inferior.

This aqueous suspension of the pigment may then be poured with stirring into the vehicle, which may for example be boiled linseed oil or a hydrocarbon oil of the paraffin type. To the resulting emulsion I preferably add just sufficient ammonia so that the characteristic ammoniacal odor persists, and thoroughly stir the mixture.

Or, and preferably, I first emulsify the oil by stirring the same with a dilute aqueous solution of ammonia, and then introduce the pigment suspension with thorough stirring.

However the pigment-oil-water mixture or emulsion may be prepared, I now add a small proportion of any flocculating electrolyte, such for example as a solution of alum or dilute hydrochloric acid, and after stirring allow the mixture to stand until a curd-like precipitate or deposit is formed.

It will be found that these curds consist chiefly of oil and pigment from which the bulk of the water has been eliminated. The excess water may be poured off, and the remaining curds drained, as for instance by changing them into a muslin filter bag or the like. Any residual water may be evaporated off in any suitable way, as for instance by passing the mass through steam heated rolls. The resulting product is ready for packaging, and constitutes a stock material which may be compounded as desired with a variety of oily or other vehicles for the manufacture of paints, inks, varnish preparations or analogous fluid coating compositions.

I claim:

1. Method of preparing pigment-oil compositions comprising preparing an emulsion of oil and water having a pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; and recovering the resulting oil-pigment curd or composition.

2. Method of preparing pigment-oil compositions comprising preparing an emulsion of oil and water having a deflocculated pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; and recovering the resulting oil-pigment curd or composition.

3. Method of preparing pigment-oil compositions comprising preparing an emulsion of oil and ammoniacal water having a pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; and recovering the resulting oil-pigment curd or composition.

4. Method of preparing pigment-oil compositions comprising preparing an emulsion of oil and ammoniacal water having a de-

flocculated pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; and recovering the resulting oil-pigment curd or composition.

5. Method of preparing pigment-oil compositions comprising preparing an emulsion of an oil and water having a pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; recovering the resulting oil-pigment curd or composition; and eliminating residual water therefrom by evaporation.

6. Method of preparing pigment-oil compositions comprising preparing an emulsion of an oil and water having a deflocculated pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; recovering the resulting oil-pigment curd or composition; and eliminating residual water therefrom by evaporation.

7. Method of preparing pigment-oil compositions comprising preparing an emulsion of an oil and ammoniacal water having a pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; recovering the resulting oil-pigment curd or composition; and eliminating residual water therefrom by evaporation.

8. Method of preparing pigment-oil compositions comprising preparing an emulsion of an oil and ammoniacal water having a deflocculated pigment suspended therein; co-precipitating the oil and pigment by addition of an electrolyte; recovering the resulting oil-pigment curd or composition; and eliminating residual water therefrom by evaporation.

In testimony whereof, I affix my signature.

GEORGE WILSON ACHESON.

UNITED STATES PATENT OFFICE.

WILBUR CLARK KNOWLTON AND HARRY A. HOFFMAN, OF AKRON, OHIO, ASSIGNORS TO THE B. F. GOODRICH COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

COMPOUNDING RUBBER WITH POWDERY SUBSTANCES.

1,286,024. Specification of Letters Patent. Patented Nov. 26, 1918.

No Drawing. Application filed August 26, 1917. Serial No. 188,898.

To all whom it may concern:

Be it known that we, WILBUR CLARK KNOWLTON and HARRY A. HOFFMAN, citizens of the United States, residing at Akron, in the county of Summit and State of Ohio, have invented certain new and useful Improvements in the Art of Compounding Rubber with Powdery Substances, of which the following is a specification.

This invention relates to the art of compounding a plastic gum such as rubber, requiring considerable mastication for its incorporation with filling materials, with substances such as gas-black (a species of lampblack produced from natural gas) normally existing as an impalpable powder, our object being to keep the atmosphere in the vicinity of the mixing mill substantially free from the dust or powder, thus preventing waste and enhancing the comfort of the operatives. Instead of using the lampblack in its natural condition, we prepare and treat the fine powder so as to cause its concretion into friable grains or small lumps, dry and substantially free from dust, and in this form incorporate it with the rubber on the roller mill or in any suitable masticating apparatus heated in the usual way to soften the rubber, the friability of the lumps or grains permitting a uniform distribution of the filler throughout the rubber. We may use water or other volatile liquid alone as a concreting agent, first mixing enough of it with the lampblack to produce the desired condition, and then drying the mass before mixing it with the rubber, or we may use a liquid binder composed of water and a small quantity of a soluble agglutinant such as glue, starch, dextrose or sugar, or some binder insoluble or only partly soluble in water, but miscible therewith and having a tendency to hold the particles of the material together in the desired form, the binder remaining in the lampblack after evaporation of the water but not being present in sufficient quantity injuriously to affect the qualities of the rubber compound.

By way of example, we may take by weight, 240 parts of lampblack, 1000 parts of hot water and 5 parts of starch, the starch being first dissolved in the water and the solution then thoroughly mixed with the lampblack, after which the resulting paste or syrup is dried in shallow evaporating pans and the material is then ready for use. These proportions, however, may be varied within rather wide limits.

We do not claim broadly the granulation of the powder with a volatile liquid before mixing with the rubber, our invention being an improvement upon the process of that nature described in application of William C. Geer, Serial No. 100,153, filed May 26, 1916.

We claim:

1. The method of compounding a plastic substance with an impalpable powder which consists in causing a concretion of the powder into dry, granular or lumpy form, and then mixing it with the plastic substance.

2. The method of compounding rubber with a substance normally existing as an impalpable powder which consists in wetting the powder and drying it to produce a friable, granular or lumpy condition, and incorporating the dry, granular substance with the rubber.

3. The method of compounding rubber with lampblack which consists in wetting the powdered lampblack to concrete it, and drying the same, bringing it to a condition of friable grains or lumps, and incorporating the dry, granular lampblack with the rubber on a heated mixing mill.

4. The method of compounding rubber with lampblack which consists in mixing the lampblack with water, evaporating the water, leaving the lampblack in a granular or lumpy condition, and then mixing it with the rubber.

5. The method of compounding a plastic substance with an impalpable powder which consists in mixing the powder with a concreting and binding liquid, drying the same, producing a condition of friable grains or lumps, and mixing it with the plastic substance.

6. The method of compounding rubber with lampblack which consists in mixing the lampblack with water and a binder, evaporating the water, producing a condi-

tion of friable grains or lumps, and mixing said grains or lumps with the rubber.

7. The methed of compounding rubber with lampblack which consists in mixing the lampblack with water and a binder, producing a granular condition, evaporating the water, and incorporating the dry, granular lampblack with rubber on a heated mixing mill.

In testimony whereof we have hereunto set our hands this 24th day of August, 1917.

WILBUR CLARK KNOWLTON.
HARRY A. HOFFMAN.

UNITED STATES PATENT OFFICE.

WILLIAM C. GEER, OF AKRON, OHIO, ASSIGNOR TO THE B. F. GOODRICH COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

COMPOUNDING OF RUBBER.

1,245,700. Specification of Letters Patent. Patented Nov. 6, 1917.

No Drawing. Application filed May 24, 1916. Serial No. 100,182.

To all whom it may concern:

Be it known that I, WILLIAM C. GEER, a citizen of the United States, residing at Akron, in the county of Summit and State of Ohio, have invented certain new and useful Improvements in the Compounding of Rubber, of which the following is a specification.

This invention relates to the compounding of rubber in a mixing mill with ingredients such as gas-black which occur in the form of a powder so finely divided or impalpable that if used in a dry condition they tend to spread through the atmosphere of the mill room, causing the loss of a portion of the powder and discomfort to the operatives.

I find that by mixing the powder with a suitable liquid before it is introduced to the batch of rubber on the rolls of the mill, and bringing the powder to a granular condition through the wetting of the particles and their concretion into grains, the objection stated is entirely overcome.

Two liquids which I have successfully employed for this purpose are water and kerosene oil, both of which have the effect of rendering the gas-black dustless and yet not pasty when mixed therewith in suitable proportions. For example, I mix two parts, by weight, of gas-black with one part of kerosene oil in a suitable closed vessel which preferably contains rapidly-revolving paddles which assist the mixing and promote the attainment of the granular condition. The oil coats and concretes the particles as stated and brings the mass into a condition suitable for its proper distribution throughout the rubber on the heated rolls of the mixing mill. The volatility of the oil is such that a portion of it evaporates from the batch on the rolls and substantially all of the residue evaporates from the finished vulcanized articles made from the rubber batch within a few days after the article is cured, this being shown by the gradual increase in tensile strength of the vulcanized rubber until within a few days its strength becomes normal. Pneumatic tire treads compounded in this way show no difference in wear from those compounded with dry gas-black.

The use of water gives an effect substantially the same as kerosene, but it requires a longer time for compounding on the mill in order to permit the drying-out of the water.

I claim:

1. The method of compounding a fine powder with rubber on a mixing mill which consists in wetting the powder and then introducing it to the batch of rubber on the mill.

2. The process of compounding rubber with an impalpable powder which consists in rendering the powder granular and substantially dustless with a liquid and then compounding it with the rubber.

3. The method of compounding a plastic such as rubber with an impalpable powder such as gas-black which consists in wetting the powder with a volatile liquid which is subsequently evaporated, to render the same substantially dustless, and incorporating said substantially dustless powder with the rubber.

4. The method of compounding rubber with gas-black which consists in wetting the gas-black with a volatile liquid which is subsequently evaporated, and incorporating the same with the rubber on a mixing mill.

5. The process of compounding rubber with an impalpable powder which consists in bringing the powder with oil to a dustless granular condition and then compounding it with the rubber.

6. The process of compounding rubber with an impalpable powder which consists in bringing the powder with kerosene oil to a dustless granular condition and then mixing it with the rubber.

7. The process of compounding rubber with gas-black on a mixing mill which consists in bringing the gas-black with kerosene oil to a dustless granular condition and then compounding it with the rubb

In testimony whereof I hav hereunto set my hand this 10th day of May, 1916.

WILLIAM C. GEER.

AMENDED SPECIFICATION.

Reprinted as amended in accordance with the decision of the Assistant-Comptroller (Patents), dated the 29th day of February, 1924.

PATENT SPECIFICATION

Convention Date (United States): July 20, 1921. **183,430**

Application Date (in United Kingdom): May 31, 1922. No. 15,336/22.

Complete Accepted: March 22, 1923.

COMPLETE SPECIFICATION (AMENDED).

Improvement in Process of Agglomerating Fine Materials.

We, TRENT PROCESS CORPORATION, a corporation organized under the laws of the State of Delaware, doing business as Manufacturers at 1440, U Street, in the City of Washington, District of Columbia, United States of America, Assignees of WALTER EDWIN TRENT, of Wardman Park Hotel, Washington, D.C., U.S.A., a citizen of the United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to a process of treating finely divided carbonaceous material of the kind in which the material in liquid suspension, is agitated and subjected to the action of a hydrocarbon treating agent, whereby the carbonaceous material agglomerates with the agent in an agglomerate which excludes the ash forming impurities and water. It is well known that all coals contain in varying proportions besides a carbonaceous and hydrocarbon content, ash forming materials commonly known as gangue, consisting of clay, silica, *etc.*, which constituents interfere with the efficient use of the coal by forming clinker. Walter E. Trent's Patent Specification No. 151,236 discloses a process for removing a large percentage of the impurities referred to, wherein the fine particles are suspended in water and agitated in the presence of the hydrocarbon oil which oil serves to combine with the fine particles in one dense mass or in a plurality of variable sized masses which are troublesome to remove because they will not be carried by the flowing water. The exact nature of the reaction whereby the carbonaceous material is separated from the impurities referred to is not entirely understood at present. Possibly it results from the non-miscibility of the water and liquid hydrocarbon, and the greater affinity between the liquid hydrocarbon and the carbon particles than that between said liquid and the ash forming particles or between the carbon particles and water. However this may be, it is clear that if a mass of carbonaceous material suspended in water is treated with a suitable hydrocarbon agent in suitable quantity as set forth in the Specification No. 151,236 aforesaid, the oil possesses characteristics such as to expel the water and to agglomerate or collect the carbonaceous matter.

We are aware that it has been proposed in froth flotation processes to dispose the froth concentrate on a screen so that the free water may be drained off.

It is an object of this invention to provide a process for forming these agglomerates of the fine carbonaceous particles and oil of substantially uniform size, which shall be of substantially spherical form, which agglomerates are capable of being carried by the water in which they are contained to a point of collection and there separated from the water and suspended impurities. Our invention is primarily based on the discovery that by controlling the character of agitation and the period of treatment of the mixture consisting of fine comminuted carbonaceous particles with water and a proper proportion of hydrocarbon oil, said particles combine in aggregates of a spherical form of a diameter ranging from $^1/_{16}$ of an inch to 2 inches, and when so formed can be

[*Price* 1/-]

discharged with the water from the agitator, because of the fact that they are carried by the moving water in which the fine particles were originally suspended and discharged as independent aggregates upon a suitable collector, preferably a screen through which the water passes with the ash forming substances.

According to this invention the process consists in placing the finely divided carbonaceous material in liquid suspension, applying to the material an agent such as oil, and agitating the substances under such conditions as to cause the agent to become associated with the fine particles and to unite said particles into a plurality of independent and substantially spherical aggregates largely freed of the liquid and ash forming impurities and of such a substantially uniform size as to be capable of being carried by the moving liquid, and removing said independent aggregates from the agitating receptacle by means of the flow of the suspension liquid.

The suspension liquid (water), the finely divided carbonaceous material and the oil are preferably continuously supplied to the first of a series of compartments, and the agglomerated or aggregated oil and carbonaceous material are carried from one compartment to a succeeding compartment by a continuous current of water by which the independent aggregates are delivered out of the last compartment.

To practice successfully the process each particle of material should be agitated at a certain degree of violence to form the spheres and a definite time of treatment to control the size of spheres or aggregates formed. The longer the period of treatment the larger the size of the aggregate.

The method is adaptable either to intermittent batch treatments or to a continuous operation so long as the type of treatment is applied so as to form the spheres capable of being carried by the water from the agitator. The preferred manner of carrying out the process comprises a continuous operation in which we mix in water suspension a quantity of finely powdered coal. The material is then passed through an agitating chamber into which is also introduced a proper amount of hydrocarbon oil or other agglomerating agent, said agitating chamber being so constructed that the period of time of treatment of each particle during passage therethrough is definitely controlled, as well as the violence and character of agitation. To regulate the time of treatment of the fine material many types of apparatus may be employed, but we have found that a single mixing chamber, divided into separate compartments in which agitation is effected in each compartment, is a satisfactory type of apparatus. With such an apparatus, the coal, water and oil are agitated at a definite rate and for a definite period in each compartment, the substances then successively passing through the succeeding compartments, and when they emerge from the last compartment the carbonaceous particles have agglomerated with the oil or other agent into aggregates or substantially spherical bodies of a substantially uniform size, ranging from approximately $^1/_{16}$ to 2 inches in diameter, and are carried by the flow of discharging water from the mixing chamber and deposited upon a receiver for separation and collection. The water with suspended impurities is thus separated from the spherical bodies of oil and coal which are of dense plastic consistency and substantially free from detached ash forming substances.

By forming the amalgamated substances, as hereinabove described, our process possesses the desirable feature of enabling the purification of the carbonaceous particles to be accomplished continuously although the treatment can be carried on intermittently in tanks or suitable receptacles, however desired. A continuous treatment is, however, usually preferable, and when such a treatment is being carried out the coal in suspension in the water is continuously introduced to an elongated receptacle divided into compartments whose inlets and outlets are so placed and of such a size as to regulate the time required for said material to pass through each compartment therein. With this material there is mixed a definite proportion of oil and the three substances are agitated at a given and determined rate, which agitation, with the time of travel of the material through the receptacle, is sufficient to cause the oil to envelop the carbon particles and to unite said particles into substantially uniform spherical masses from which detached ash forming substances and a large percentage of water are eliminated. The masses thus formed flow with the water to a point of discharge and are then collected in any approved manner.

The accompanying drawings show a single embodiment of the apparatus of the apparatus for carrying out the process but obviously many types of apparatus may be employed for accomplishing the desired ends.

A receptacle 1 has an inlet 2 for finely pulverized coal and water, said coal being

sufficiently finely ground in the presence of water to physically detach the bulk of the gangue from the carbonaceous content. An inlet 3 for oil or like treating agent also leads to the receptacle. The coal introduced to the receptacle is preferably wet ground in a suitable apparatus, and is usually of 200 mesh or finer, i.e. is sufficiently fine to pass through a screen having 200 or more meshes to the square inch. The coal and the water thus introduced to the receptacle enter the first compartment or chamber 4 therein.

It will be observed that the receptacle is divided by partitions 5, 6, 7 and 8 into a plurality of compartments 4, 9, 10, 11 and 12. These compartments or chambers may be of uniform size, or if desired in the treatment of certain coals one chamber or compartment may be larger than another. Each chamber is constructed so that a given period of time will be required for the passage of fine materials therethrough.

Passing through the receptacle is a shaft 13, having a pulley 14 and a plurality of agitating blades 15, a set of said blades being arranged in each of the compartments 4, 9, 10, 11 and 12. The materials introduced to the first compartment 4 undergo a partial commingling therein, and the rate of flow is so regulated by the outlet therefrom as to require each portion of the material to remain in this compartment the desired period of time, prior to its discharge through the opening 16 into the compartment 9, and successively into the other compartments. After desired agitation in the compartment 9, the partially commingled oil and coal carried in the water containing suspended impurities pass through the opening 17 and into the compartment 10 undergoing an agitation therein and thence passing through the opening 18, into the compartment 11 and thence through the opening 19 into the compartment 12. Before the material reaches the last compartment the oil will have enveloped and surrounded the fine particles of coal and a multitude of said particles are joined into amalgamated masses of substantially uniform size and of substantially spherical form, from which the ash content and a large portion of the water are rejected, the ash content being carried by the water to a point of discharge while the agglomerates are collected.

In the treatment of bituminous coals of 200 mesh to produce spherical bodies of agglomerated carbonaceous particles and oil of uniform size of approximately onehalf inch diameter, the agitator may be revolved at a speed of approximately 125 revolutions per minute, the time of agitation being approximately 50 minutes. Of course the speed of rotation of this shaft and the time of treatment largely depend on the type of agitator employed, and we have merely referred to the time of treatment and the rate of agitation for the purpose of disclosing a single type of apparatus which we have successfully employed, although we wish it to be distinctly understood that these factors are subject to wide fluctuation, depending upon various types of agitators, mixing chambers and other apparatus that may be used. The agglomerates produced in the agitating apparatus vary with different kinds of carbonaceous matter, but in the treatment of bituminous coals we have found that the agglomerates will all be of substantially uniform size and of a diameter approximately one-half an inch if treated as above described.

Each agglomerated mass consists of a multitude of the individual carbonaceous particles that have been separately coated with thin films of oil. The agglomerates are, as before stated, largely free from detached ash, and contain little moisture, usually ranging from 10 to 15 *per cent.* In the treatment of anthracite the agglomerates are usually somewhat smaller, but are of spherical form and of a diameter of approximately one-fourth of an inch. The coal and oil introduced to the receptacle 1 must be in regulated and definite quantities and we preferably introduce to said receptacle a hydrocarbon oil, such as fuel oil in the proportion of 1 part by weight of oil to 2 parts by weight of carbonaceous content of the coal undergoing treatment. The quantity of water is not a serious factor, although we preferably use sufficient water to place the coal in free suspension.

From the receptacle 1 the agglomerated masses are carried by the water through the discharge trough 20 and deposited upon an endless travelling screen 21. This screen is mounted upon the rotary supports 22 and the agglomerated masses as they fall on the screen are moved forward and discharged into a receptacle 23. The water, with the suspended impurities, flows through the screen into the trough 24 being conveyed by the pipe 24¹ to a settling receptacle 25, or allowed to escape if this is desired. In the settling receptacle 25, however, the ash forming substances collect in the bottom, and the water may then be again used in the treating apparatus. Any oil that may happen to escape with the water is also recovered for reuse.

During the passage of the amalgamated masses on the screen 21 from the trough

20 to the receptacle 23 we preferably subject these masses to the action of a cleaning spray by directing jets of water fed through a pipe 26 through the bottom of the screen and into contact with the amalgamated masses. This serves to wash away some surface impurities, and cleanse the product. We like wise direct a water spray downwardly through the underside of the screen 21 for the purpose of cleaning said screen of any substances which may be contained thereon so that a clean screen surface is presented to receive the discharging amalgam.

Experience has shown that in treating certain kinds of coals, it is sometimes desirable to introduce the oil, in part at the point of introduction of the coal or other carbonaceous matter and in part at another point or other points along the line of travel of the coal through the agitating receptacle.

In other processes where hydrocarbon agents have been used for the purpose of flocculating coal so as to render it readily separable from water, it has been proposed to use a small proportion of the agent, such as 5 or 7 *per cent*, but according to the present invention sufficient hydrocarbon is used to fill up the spaces between the coal particles in the globular aggregate to the substantial exclusion of water.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process of treating finely divided carbonaceous material according to the process disclosed in the Specification of Patent No. 151,236, which consists in placing the material in liquid suspension, applying to the material an agent such as oil, and agitating the substances under such conditions as to cause the agent to become associated with the fine particles, and to unite said particles into a plurality of independent and substantially spherical aggregates largely freed of the liquid and ash-forming impurities and of such a substantially uniform size as to be capable of being carried by the moving liquid substantially as described and shown in the accompanying drawing.

2. A process according to Claim 1 in which the treatment is continuously effected in a series of compartments and the agglomerated or aggregated carbonaceous material and oil are carried from one compartment to a succeeding compartment by a continuous current of water, by which the independent aggregates are delivered out of the last compartment, substantially as described and shown in the acompanying drawing.

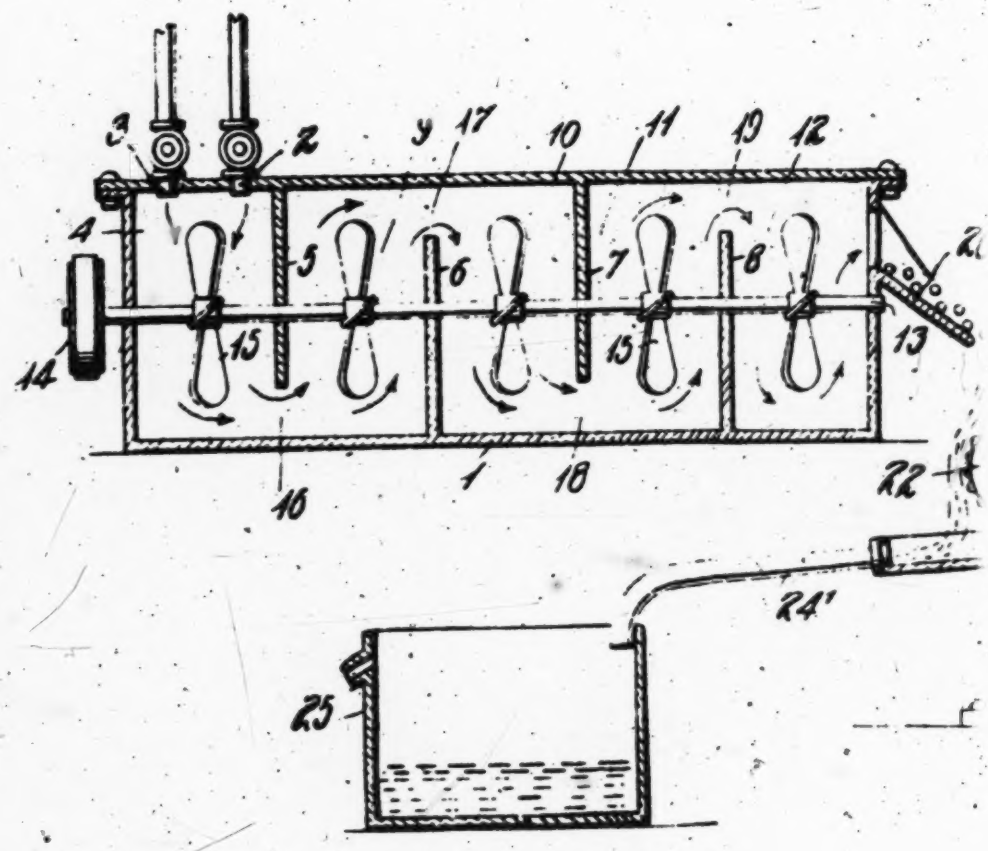
3. In a process according to Claim 1, separating the independent aggregates from the water in which they are contained substantially as described and shown in the accompanying drawing.

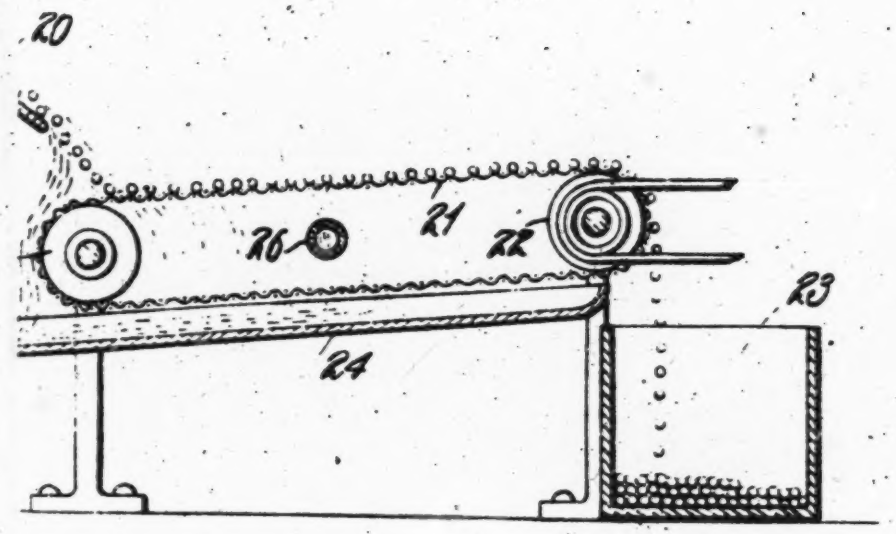
Dated this 31st day of May, 1922.

For the Applicants,
HERBERT HADDAN & Co.,
Chartered Patent Agents,
31 and 32, Bedford Street, Strand,
W.C. 2, London.

Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.—1924.

[This Drawing is a full size reproduction of the Original]





V Powers

Portion of Defendant's Exhibit 23. 317

Nov. 17, 1925. 1,561,971

J. G. COFFIN ET AL

METHOD FOR TREATING PULVERULENT MATERIAL AND PRODUCT

Filed Oct. 16, 1923 2 Sheets-Sheet 1

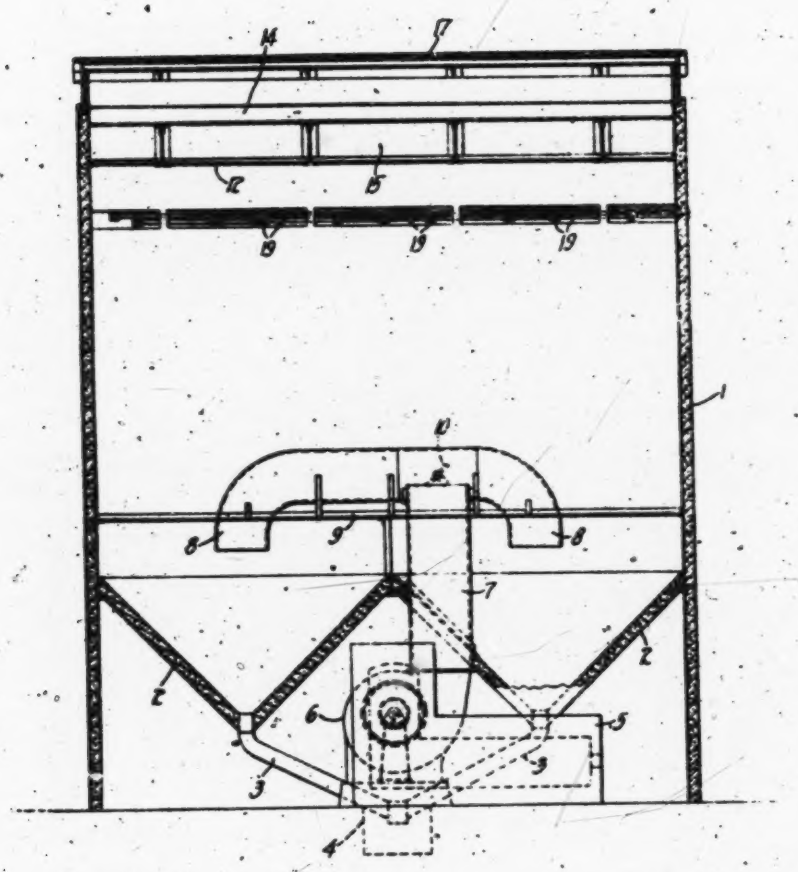


Fig. 1.

Inventors
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By their Attorney
Ernest Hopkinson

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Nov. 17, 1925. 1,561,971

J. G. COFFIN ET AL

METHOD FOR TREATING PULVERULENT MATERIAL AND PRODUCT

Filed Oct. 16, 1923 2 Sheets-Sheet 2

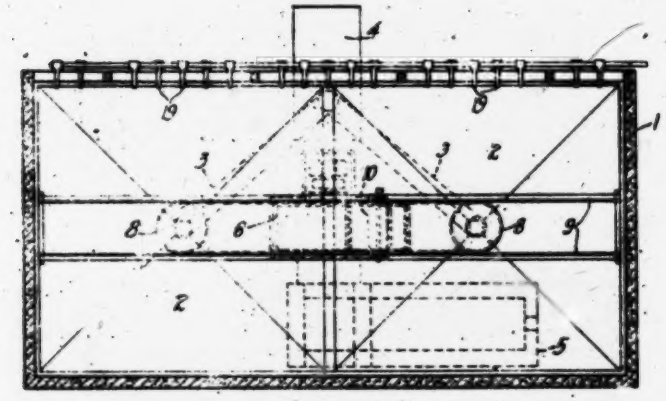


Fig. 2

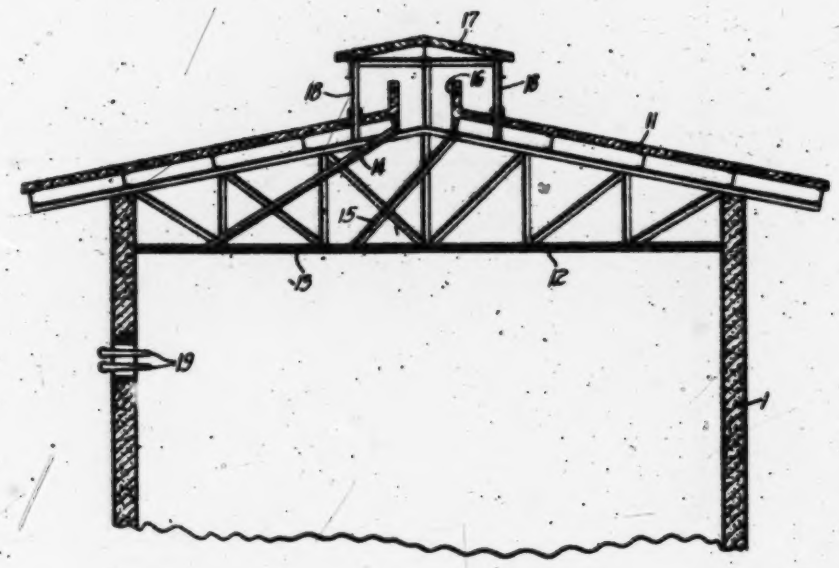


Fig. 3.

Inventors
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ALEXIS W. KEEN
By their Attorney
Ernest Hopkinson

Patented Nov. 17, 1925. 1,561,971.

UNITED STATES PATENT OFFICE.

JOSEPH G. COFFIN, OF HEMPSTEAD, AND ALEXIS W. KEEN, OF NEW YORK, N. Y., ASSIGNORS TO THE NAUGATUCK CHEMICAL COMPANY, OF NAUGATUCK, CONNECTICUT, A CORPORATION OF CONNECTICUT.

METHOD FOR TREATING PULVERULENT MATERIAL AND PRODUCT.

Application filed October 16, 1923. Serial No. 668,867.

To all whom it may concern:

Be it known that we, JOSEPH G. COFFIN and ALEXIS W. KEEN, both citizens of the United States, and residing at Hempstead, Long Island, in the county of Nassau and State of New York, and New York city, county of New York, and State of New York, respectively, have invented a certain new and useful Method for Treating Pulverulent Material and Product, of which the following is a full, clear, and exact description.

This invention relates to a method for treating pulverulent material and to the product thereof, and more particularly to a method for directly drying clay in suitable form, and to the product.

In addition to its extensive use for pottery and various other purposes in the arts, clay has more recently been extensively used as a filler for rubber compounds. When used for such purpose the clay must be free from grit and moisture and in such form that it can be readily incorporated with the rubber mix on a mill. By the method at present in use the clay after first being mixed with water to form a so-called "slip", is treated to remove grit and large particles and some of the water, and it is then conveyed to a filter press in order to remove the bulk of the remaining water. From the filter press the wet clay is taken to drying racks on which it is disposed in lumps and subjected to heat. The dried lump clay is then stored or shipped to the place where it is to be further treated or used. Before the lump clay can be used for many purposes, such as for instance in a rubber mix, it must be subjected to a second drying operation and then pulverized on a mill, after which the powdered clay is placed in storage or conveyed to the place where it is to be used. The above operations require a considerable expenditure of time, labor, space and apparatus, and there is an objectionable dusting in handling the dried material.

An object of our invention is to provide a rapid and economical method for drying pulverulent material such as clay.

Another object is to provide a method for directly drying clay and similar material in subdivided form.

A further object is to provide a relatively fluent dry pulverulent material, and

A still further object is to provide a dry clay or similar material in a fine granular form.

The method comprises broadly treating a pulverulent material such as clay in fluid suspension to remove large particles therefrom and subsequently to reduce its fluid content, and then finely dividing the treated suspension to form it into small globular masses which are then dried in the form of small globules of lightly cohering particles of the material while in suspension in a gaseous drying medium. The invention also consists in the product of the method and apparatus.

For a detailed disclosure of the invention reference is had to the accompanying specification and drawings illustrating one form of apparatus for carrying out the invention, in which drawings:—

Figure 1 is a part central longitudinal section through the apparatus for carrying out the method,

Figure 2 is a horisontal section thereof, and

Figure 3 is an enlarged transverse section of the upper part of the apparatus taken at right angles to that in Figure 1.

The method will first be described in its application to the treatment of clay, but it is suitable for the preparation of any other pulverulent material in a conveniently handled and desirable form.

In carrying out the invention the clay, if not already in the form of a "slip" through hydraulic mining, is mixed with water to form a suspension, and the larger particles such as grit, pebbles, etc., removed by settling. The "slip" is then allowed to further settle to remove a part of the water, and the thickened but still fluid clay is pumped to the drying apparatus shown in Figs. 1 to 3. This apparatus in the present instance consists of a heat insulated casing 1 forming a drying chamber. The casing as shown consists of inner and outer sheet metal walls having any suitable insulating material disposed between, but any other desirable construction may be used such as concrete or hollow tile. The bottom as shown preferably consists of hoppers 2 in the form of inverted pyramids, the number of which will be varied in accordance with the size of the drying chamber. The slant of the hopper bottoms is made sufficient to

allow of the ready discharge of the dried material, which then flows through the pipes 3 to a collecting receptacle 4.

Any suitable drying medium may be used in the drying chamber, but in the present instance that used is a mixture of air and combustion products supplied by the furnace 5. The furnace is preferably of the general type shown in the patent to Bradley and Coffin, No. 1,466,443, Aug. 28, 1923, except that the air mixing chamber is disposed transversely on top of the combustion chamber instead of at the side thereof, but any other suitable form of furnace may be used. The mixed combustion products and air are withdrawn from the furnace by the fan 6 and conveyed through the upright pipe 7 to the downwardly turned nozzles 8, which latter are preferably disposed centrally above the hoppers 2. The nozzles 8 may be rigidly held in position by any suitable means such as the angle irons 9. In order to properly regulate the flow of the drying medium to the nozzles 8 a small deflecting damper 10 is provided.

At its top the casing 1 is provided with a roof 11, below which is disposed a ceiling 12 forming the top of the drying chamber. Adjacent one side of the chamber the ceiling is provided with an elongated exhaust opening 13, preferably extending the entire length of the chamber, which communicates with an inclined passage-way formed by the walls 14 and 15, this passage-way discharging at its upper end through an opening 16 in the roof within the space below a monitor 17, from which space the exhaust products escape into the atmosphere through the openings 18. If desired the ceiling 12 and inclined passage-way may be omitted, in which case the exhaust opening will be formed in the roof and open directly into the monitor 17, but this exhaust opening will of course be disposed in about the same relative position with respect to the spray nozzles as previously. Adjacent the top of the drying chamber and on the same side as the exhaust opening 13 a plurality of spray nozzles 19 are disposed in horizontal series, the number and arrangement of which nozzles may be varied to suit conditions.

In carrying out the method the clay, which has been purified and partly freed of water as before described, is supplied to the nozzles 19 and sprayed transversely into the drying chamber. The globular sprayed particles in their descent come in contact with the ascending current of the drying medium discharged from the nozzles 8 and the dried material deposits as very small globular masses or pellets on the sides of the hoppers 2, while the exhaust drying medium escapes through the opening 13 and thence to the outer air. It will be seen that the exhaust opening, which extends the entire length of the drying chamber, is disposed on the same side of the latter as the spray nozzles 19, hence the drying medium is compelled to pass through the spray discharged by the nozzles 19 before it can escape, and all light dried or partially dried material that might otherwise escape with the exhaust is retained by the wet spray. By reason of the arrangement of the nozzles 8 the drying medium is discharged downwardly into the hoppers 2 and then sweeps upwardly along the walls of the hoppers and into the main body of the drying chamber. The drying medium is introduced at a rather high temperature, about 500° to 700° F. for instance, and as a result of the described construction contacts while at its highest temperature with the clay globules rolling down the hopper bottoms or about to deposit thereon, thereby producing a very thoroughly dried product. The dried clay as before stated deposits in the hoppers in the form of very small friable globular masses or pellets, each of which is an aggregate of lightly cohering particles of clay, and the product very much resembles a fine sand in its appearance to the naked eye. Hence it does not tend to compact when in bulk but will flow very readily, and therefore, easily rolls to the bottoms of the hoppers 2 and through the discharge pipes 3 to the receptacle 4. The material may be conveyed by any suitable means from the receptacle 4 to a bagging apparatus or disposed for storage in any suitable manner. Due to the rounded granular form in which the clay is obtained and the absence of any sharp particles it is extremely fluent and, therefore, can be very readily handled as compared with the compact mass formed when clay is in a pulverized state. This property of fluency is so marked that if desired the hopper bottoms 2 may be omitted and a flat bottom substituted, the latter being provided with openings radially below the nozzles 8. The dried material will then accumulate in the form of mounds with slopes having a relatively small angle of repose, the succeeding material then rolling down the slopes and into the openings. In other words the accumulated dried material itself will take the place of the hopper bottoms 2. However, the hopper bottom is preferred, as there is then no accumulation of the dried material in the drying chamber, which might be objectionable under certain circumstances. As before stated, the dried grains or pellets are very small and the individual particles of clay forming them are only lightly held together, so that a very slight pressure such as that of the fingers is sufficient to crush the grains, but the bond between the particles is sufficiently strong to substantially prevent their breaking up during handling. Hence the product is en-

tirely suitable as obtained from the drying chamber for use directly in the rubber mill. Due to its form the objectionable dusting which occurs in the mixing and handling of finely powdered materials is also avoided.

It will be seen that by our invention clay is directly obtained in a dry friable form suitable for use in a rubber mill or for any other purpose with a minimum expenditure of time, labor, space and apparatus, and in particular the filtering step, one drying step, and the pulverizing step of the previously known method have been eliminated, as well as the necessary apparatus for carrying them out.

The invention is widely applicable for efficiently drying any pulverulent material, such as other rubber fillers, which is desirably ground wet in a fluid in which it is insoluble, or in which objectionable dusting occurs in handling. Examples of materials for which our invention may be used are, whiting, barytes and lithopone. The invention is also applicable where a mixture of finely divided materials is required; for example in the pottery art when several clays are to be combined they may be mixed in fluid suspension and then dried by our method.

While a specific embodiment of the invention has been shown and described it is obvious that numerous modifications may be made therein in view of the disclosure, and it is not desired to limit the invention otherwise than as set forth in the appended claims.

Having thus described our invention, what we claim and desire to protect by Letters Patent is:

1. The method of treating clay which comprises forming a fluid suspension thereof, projecting the suspended clay in finely divided form into a heated drying medium, and drying while in suspension in the form of minute globular masses composed of lightly cohering particles of clay.

2. The method of treating clay which comprises forming a slip thereof, partially removing water from the clay to form a thickened but fluid suspension, spraying the thickened clay into a heated drying medium, and drying while in suspension into the form of very small globular friable masses of lightly cohering particles of clay.

3. The method of treating clay which comprises forming a slip thereof, partially removing water from the clay by settling to form a thickened but fluid suspension, spraying the thickened clay into a moving counter current of a highly heated drying medium, and drying while in suspension into the form of very small globular masses of lightly cohering particles of clay.

4. The method of treating pulverulent material which comprises forming a suspension thereof in a fluid in which it is insoluble, sub-dividing said suspension into very small globules, and drying in the form of very small globular friable masses composed of lightly cohering particles of the material while in suspension in a heated gaseous drying medium.

5. The method of treating pulverulent insoluble inorganic materials, which comprises forming a fluid suspension of the material, subdividing the same into small globules, and drying in the form of very small substantially globular masses of lightly cohering particles of the material while in suspension in a highly heated gaseous medium, said medium being first applied to the most nearly dried globules.

6. The method of treating pulverulent insoluble inorganic materials, which comprises forming a fluid suspension of the material, spraying said suspension into the form of small globules, and drying the globules to the form of very small substantially globular masses of lightly cohering particles of the material by passing a highly heated current of a drying medium through the spray, said current first mingling while at its highest temperature with the dryest portion of the spray and leaving the spray at the wettest portion of the latter.

7. The method of treating pulverulent water insoluble inorganic material which comprises forming a thick water suspension of the material, sub-dividing said suspension into the form of small globules, and drying said globules into the form of very small substantially globular friable masses composed of light cohering particles of the material while in suspension in a highly heated gaseous medium, said gaseous medium being first directed against the most nearly dried material and then in a direction generally counter to the direction of movement of the suspended globules.

8. As a new article of manufacture, dried clay in the form of very small approximately spherical friable pellets.

9. As a new article of manufacture, a fluent dried clay in the form of very small rounded individually dried friable pellets.

10. As a new article of manufacture, dried pulverulent material in the form of very small individually dried friable globular masses composed of lightly cohering particles of the material.

11. A fluent dried pulverulent inorganic material derived from a fluid suspension of the material by spray drying and composed of very small friable substantially globular masses of the material.

Signed at New York, in the county and State of New York, this 10th day of October, 1923.

JOSEPH G. COFFIN.
ALEXIS W. KEEN.

322 ✓ DEFENDANT'S EXHIBIT 24.

300

DEPARTMENT OF COMMERCE
UNITED STATES PATENT OFFICE

To all persons to whom these presents shall come, Greeting:

THIS IS TO CERTIFY that the annexed is a true copy from the records of this office of the File Wrapper and Contents, in the matter of the

Letters Patent of

William B. Wiegand and
Louis J. Venuto,

Number 1,889,429, Granted November 29, 1932,

for

Improvement in Carbon Black and Processes of Making Same.

IN TESTIMONY WHEREOF I have hereunto set my hand and caused the seal of the Patent Office to be affixed, at the City of Washington, this sixteenth day of November, in the year of our Lord one thousand nine hundred and forty and of the Independence of the United States of America the one hundred and sixty-fifth.

ATTEST:

Chief of Division.

Commissioner of Patents.

U. S. GOVERNMENT PRINTING OFFICE 16—11388

323

DEC 1924 U.S. PATENT OFFICE

74 Claims

PETITION

237330

TO THE COMMISSIONER OF PATENTS:

YOUR PETITIONERS, WILLIAM B. WIEGAND and LOUIS J. VENUTO, the former a citizen of Canada and a resident of Sound Beach, in the County of Fairfield and State of Connecticut, and the latter a citizen of the United States and a resident of New York, in the County of Queens and State of New York, both of whose Post Office address is c/o Binney & Smith Company, 41 E. 42nd Street, New York City, pray that Letters Patent may be granted to them for the improvements in

CARBON BLACK AND PROCESS OF MAKING SAME

set forth in the annexed specification; and they hereby appoint DEAN, FAIRBANK, CBRIEHT & HIRSCH, a firm composed of GEORGE C. DEAN, CLAIR W. FAIRBANK, IRVING M. CBRIEGHT AND MORRIS HIRSCH (Registered No. 11551) of Graybar Building, 420 Lexington Avenue, New York City, State of New York, their attorneys, with full power of substitution and revocation, to sign the drawings, to prosecute this application, to make alterations and amendments therein, to receive the patent, and to transact all business in the Patent Office connected therewith.

William B. Wiegand

Louis J. Venuto

SPECIFICATION

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT WE, WILLIAM B. WIEGAND and LOUIS J. VENUTO, the former a citizen of Canada and resident of Sound Beach in the County of Fairfield and State of Connecticut, and the latter a citizen of the United States and resident of New York, in the County of Queens and State of New York, have invented certain new and useful improvements in CARBON BLACK AND PROCESS OF MAKING SAME, of which the following is a specification:

The important characteristics of carbon black which render it particularly valuable in the manufacture of rubber compositions, phonograph records, various wax and resin compositions, printers ink and other preparations, are its extremely fine state of subdivision together with the very essential property of being readily and uniformly dispersed in rubber, waxes, oils and the like. Carbon black, as manufactured, is such a very light fine powder that normally ten pounds, or less, occupy a package of one cubic foot. It is accordingly expensive to pack, ship and store such a light bulky material, and handling the light powder causes it to fly in the air to such an extent that there is substantial loss of material, and an annoyance to workmen handling the material and breathing the dust.

Numerous attempts have been made to overcome these objectionable properties of carbon black and render it denser and less dusty, but all such attempts heretofore have greatly changed the very essential physical properties of the carbon black which make it industrially valuable. Thus, the carbon black may be made into a paste with water or other volatile liquid such as gasoline, solvent naphtha, xylene, turpentine, carbon tetrachloride and the like, and the volatile liquid then distilled or evaporated, but the resulting cake is hard, the carbon particles tend to hold together in chunks, and they no longer disperse readily and uniformly as for example in rubber compositions. Apparently the state of aggregation of the carbon particles has been changed in such altered carbon blacks, and, as a matter of fact, the industrially essential physical properties, namely, easy and uniform dispersion, are so much impaired that such processes have never been a commercial success.

We find that when the original carbon black is added to a liquid to form a paste and the liquid slowly distilled or evaporated, such treatment causes changes which are associated with a free meniscus, ie. vortices, surface tension adhesion, settling, volume contraction of the black, and a very persistent adhesion of particle to particle. The result is the clumping together of the carbon and the loss of dispersibility, as referred to above. This change of colloidal properties or loss of dispersibility may be mitigated or avoided by drying very quickly with violent agitation, drying by quickly removing the liquid by absorbing it into the walls of a small porous container, or by displacing the original liquid by an other in such a manner as to avoid free suspension of the particles, in a liquid phase.

The main object of our invention is to secure carbon black having the desired dispersive properties, greater density, freedom from dust, freedom from gritty particles, less absorbed or occluded gases, reduced oil absorption than the ordinary powder form, and capable of considerable handling without crushing or dusting.

We have discovered that we can improve carbon black as to all or substantially all of the above mentioned properties without deleteriously affecting any of the properties essential to its satisfactory industrial use, such as softness and dispersive qualities, and by a process which includes the simultaneous or successive treatment of the carbon black with two immiscible liquids, the second liquid having greater attraction for or greater ability to wet the particles.

This process, if carried out under certain conditions, causes the carbon black to form into pellets

which are hard enough to stand any ordinary shipment or handling without dusting, flying or breaking down, and which at the same time are easily crushed by moderate pressure, as between the fingers or by the pressures commonly employed in the rolls of rubber compounding machinery, printers ink mixers and the like. The crushed particles have substantially their original softness and the material disperses freely without leaving any particles of undispersed carbon in the material.

While the pellet form is a very convenient form of the carbon black, the shape of the particles is not the most important characteristic of this novel carbon black. The properties enumerated above may be incorporated into the black in the form of flakes or the pellets or flakes may be crushed and pressed into blocks without seriously affecting its valuable dispersive properties.

In carrying out the process we mix the carbon black with one liquid and add a second liquid which is substantially immiscible with the first and which has greater ability to wet the carbon particles. The mixture is then agitated until the first mentioned liquid is practically free from carbon. This liquid is then separated and the other liquid is removed by evaporation.

Merely as an example of our improved process, the operation may be carried out as follows:

The carbon black is first mixed with water so as to form a comparatively thin paste with most of the occluded gases driven out. After thorough mixing gasoline is added and the whole mass thoroughly agitated. As a result substantially all of the carbon black floats on top of the water in the form of pellets and may be readily removed by filtering, decanting or the like. These pellets may then be dried to remove the absorbed gasoline and such moisture as there may be contained therein. Care should be taken to insure the complete wetting of the particles with the water, as otherwise the particles will be wet directly by the gasoline and in drying harsh particles may result. In the drying operation the carbon black retains its globular, ball-like or pellet form.

On the first addition of gasoline to the black and water paste, with agitation, the mass seems to thicken like an emulsion; then on further shaking, it seems to flocculate into granules; as the agitation goes on, a slight creaming to the top of the pellets or a scum takes place, then gradually the pellets form and float to the top. The end point is reached when the water below is free of carbon - clear.

The pellets are then screened off by any of the various methods, placed in an oven or a recovery apparatus and freed from solvent and water. It has been found that

by use of this method the drying has little or no harmful effect upon the particle. Low temperature drying, and high temperature drying (around 400° Fahrenheit) leave the particle in a good soft condition. There are however various factors which will influence this.

If there is no organic residue decomposed by heat, there will be no binding of the particle. If there is, a harshness will result due to the decomposition or gumming of the residue. If too much gasoline is used, the particles will compact to comparatively hard pellets. If there is too much water and not enough gasoline, an inferior product will result.

The control however of the above process does not necessarily have to be delicate. A rather wide marg'n of safety exists.

Merely as an example of the relative proportions which we have found to operate satisfactorily, we give the following by weight:

Carbon 5 parts
Water 40 to 80 parts
Gasoline 7 to 9 parts

In carrying out our improved process with these proportions, the mass may be thoroughly shaken or otherwise agitated for about five minutes and the resulting pellets will be of about one-sixteenth of an inch in diameter and will be fairly uniform in size.

The pellets are very porous, of substantially spherical or globular form, have a smooth somewhat lustrous outer surface which is not easily broken by handling, are more compact than untreated carbon, are fragile under light pressure, and may be easily reduced to soft minute particles which cannot be told from the original particles except that possibly they have a more unctuous feel. They somewhat resemble lead shot and may be rolled in the hand without dirtying or dusting. Apparently the outer surface portion or shell of each pellet is slightly more compact than the inner part, but still porous.

In shipping or storing, we find that approximately twice the number of pounds of these pellets can be placed in a container of a given size than is the case with the untreated carbon black. Thus, expense is reduced for shipment or storage.

It will be noted that during the agitation and separation any grit which there may be in the carbon black may settle to the bottom, while the pellets float to the top and thus the purification of the carbon black will be effected at the same time the pellets are formed.

The pellets will not pack down in the bottom of a bag or other shipping container as in the case with untreated black and may be stored in bins and delivered through chutes to the point of use, without caking or sticking. The non-flying properties permit better control of the mixing in rubber or other composition, facilitate weighing, result in a substantial saving of material by eliminating mechanical loss, and add to the comfort of the operator.

The process of the present invention when carried out as described above yields substantially pure carbon black, but we find that the present invention can be employed for the manufacture of carbon black containing other materials uniformly distributed through or absorbed by the black, without loss of dispersibility. For example in its application in the rubber industry, it has been attempted to incorporate various other substances with the black, such as accelerators, softeners and anti-oxidants. When this is done by adding the said additional substance and the carbon black to a liquid in which the added ingredient is soluble, and subsequently evaporating or distilling the liquid, the resulting carbon black is hard and has lost its ready dispersibility, as noted above. We find that the addition of substantial proportions of certain other substances to either of the liquids used in the process of the present invention does not impair the dispersibility of the resulting carbon black. Thus for example hexamethylene tetramine and various water soluble xanthates may be added to the water containing the carbon black or such substances as diphenyl-guanidine, thiocarbanilide, various amine oleates or stearates, or anti-oxidants, such as hydroquinone, resorcinol or naphthylamine may be added to the gasoline, benzol or water insoluble phase up to the limit of their respective solubilities, without impairing the qualities of the resulting carbon black.

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There are various factors which enter into the process and these may be varied to get the pellets harder or softer or larger or smaller. Among these factors are the thickness of the paste, the amount of gasoline used, the adding of the gasoline in bulk or a little at a time, speed of agitation, temperature, type of gasoline used, and character of the carbon black. In using gasoline, it is desirable to use a high grade, such for instance that commonly known as aviation gasoline, in which there is a minimum amount of high boiling constituents. Other liquids, such as benzol, chloroform, toluol, carbon tetrachloride, xylol, turpentine, or carbon di-sulphide, may be used. It is important that the liquid used be one which is as nearly completely immiscible with water as possible.

Using an agent, such as carbon tetrachloride, chloroform, carbon di-sulphide, or any other of like character having a specific gravity of over one, will not cause the flotation of the pellets to the top of the water, but they will simply sink to the bottom. They can however be recovered by filtration or screening.

Some creaming agents have greater wetting power than gasoline, and therefore require a lesser amount to bring about the end point.

In general it is preferable that the creaming agent be completely volatile so as to leave the carbon pure. In special cases it is not necessary where the residue will impart special properties to the pellet.

-0-

If small pellets are desired, a lesser amount of gasoline or other liquid should be used in respect to the amount of water and carbon, and greater agitation should be employed. To secure large pellets, we use a larger amount of gasoline and slower agitation. In practice, we do not consider a size larger than one-quarter of an inch desirable. There are many kinds, grades or varieties of carbon black and often identification of the particular kind or grade is difficult. With our improved process the different kinds or grades may be made into pellets of different sizes so that identification is facilitated, for instance, very small pellets may be made for printers ink and larger ones for rubber, etc.

It is possible also to reduce the flying of the black b not carrying the above treatment to its ultimate pellet stage. It will come out as an amorphous mass somewhat flaky and fluffy, and must be separated from the mixture by filtration

The flake form of the material is produced by incorporating the black in water, as described above, and immersing in the mixture a porous material which is wet with gasoline, or other liquid as before described. Owing to the preferential wetting power of the gasoline, the carbon particles leave the water and are wetted by the gasoline, building up a layer instead of forming globules or pellets as is the case when the agitation method is used. The surface on which the black is deposited may be a rotating porous cylinder, with the gasoline supplied to the inside, the layer of black being continuously scraped off the cylinder, as it rotates.

Another method of producing the flake form is to crush the pellets before evaporating the solvent, as between rolls, and then evaporating the gasoline, or other volatile liquid.

The whole process of pellet formation depends upon the selective wetting power which the various solvents exert on the carbon black.

We find that the present invention can be applied to other fine powders whose industrial applications depend upon their dispersibility. For example, precipitated magnesium carbonate, zinc oxide, titanium oxide, barium sulfate, and certain talcs and clays are difficult to disperse in rubber or oil on account of absorbed air and tendency to fly as dust, and we find that these powders can be manufactured in denser form, with elimination of most of the absorbed air and without loss of dispersibility, by the process of the present invention.

In the foregoing we have referred to agitation of the mixture, but we do not wish to be restricted to any particular kind of agitating apparatus. For batch operation the agitation may be accomplished by violent shaking, rotating or tumbling of the container, and for continuous operation the agitation may be by any means which will impart rapid changes in the direction and/or velocity of the flow of the mixture stream, as for instance, apertured obstructions or baffles in a conduit.

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Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. Soft carbon black particles adhering in the form of smooth surfaced porous aggregates.

2. Carbon black in the form of pellets of sponge-like or porous structure.

3. As an article of manufacture, a pellet of approximately one-sixteenth of an inch in diameter and formed of a porous mass of substantially pure carbon black.

4. A pellet formed of soft carbon black particles, the pellet being sufficiently hard to withstand ordinary shipment or handling, but readily breaking down to a fine state of subdivision upon the application of slight pressure.

5. A porous pellet comprising soft particles of carbon black.

6. A carbon black pellet formed of soft particles, the outer surface of the pellet being smooth and the outer portion being slightly denser than the inner portion.

7. A carbon black pellet formed of soft carbon black the pellet having sufficient hardness to withstand ordinary shipment or handling without dusting, but sufficiently fragile to permit reduction to the original fine state of subdivision upon the application of light pressure.

8. Soft carbon black particles cohering in small masses of substantially uniform size and having smooth outer surfaces.

9. Carbon black having substantially greater density than original carbon black, but substantially the same dispersive properties.

10. Carbon black aggregates of increased density and fragile porous structure, readily reducable to the original state of fineness, softness and dispersive properties.

11. The process of treating carbon black which includes agitating it in the presence of two substantially immiscible liquids.

12. The process of treating carbon black which includes mixing it with one liquid, and adding a second liquid which is substantially immiscible with the first, and has greater wetting power for the carbon black.

13. The method of treating carbon black which includes the steps of mixing it with one liquid to remove the occluded gas, and separating the carbon black from said liquid by the action of a second liquid substantially immiscible with the first.

14. The process of treating carbon black which includes the steps of mixing the carbon black with a liquid to form a very thin paste, adding a second liquid in smaller amount and which is immiscible with the first liquid, agitating, separating the carbon black and second

liquid from the first liquid, and then separating the carbon black and the second liquid.

15. The method of treating carbon black which consists in agitating it in the presence of two immiscible liquids, whereby the carbon black forms pellets in or with one of said liquids, separating the pellets from the liquid, and drying.

16. The process of treating a substance in a fine state of subdivision to bring it to more compact form which consists in wetting it with one liquid, agitating it with a second liquid immiscible with the first and having greater wetting properties, separating the substance from the first liquid, and drying.

17. The method of treating carbon black which includes wetting it with one liquid, and then transferring it to a second liquid having greater wetting properties, and removing the second liquid by evaporation.

18. The process of treating carbon black which includes agitating it in the presence of two substantially immiscible liquids of differing wetting power.

19. A process of treating carbon black or other fine powder comprising wetting the powder with a volatile liquid in such restricted proportion that a free menisous is not formed.

20. The method which includes wetting carbon black with water, bringing the mixture into contact with a liquid having greater wetting properties whereby the carbon black is substantially separated from the water, and evaporating the second liquid from the carbon black to leave aggregates of porous fragile structure readily redispersible to the original state of fine subdivision.

21. A process of treating carbon black which consists in agitating it in the presence of two immiscible liquids and having dissolved or incorporated in one or the other of said liquids, accelerators, softeners, anti-oxidants or other useful ingredients, whereby the carbon black forms pellets in or with one of said liquids, ~~the~~ during this process, the accelerators, softeners, anti-oxidants or other useful ingredient from one or the other of said liquids.

22. Carbon black pellets, formed of soft particles, having intimately incorporated therewith, accelerators, softeners, anti-oxidants or other useful ingredients which pellets when compounded with rubber or other substances, impart various desirable physical and chemical properties.

23. The process which includes wetting carbon black with water bringing it and gasoline into contact under conditions causing the gasoline to displace substantially all of the water from the carbon black, removing the carbon black mass wetted with the gasoline, and evaporating the gasoline.

24. The following process mixing 60 to 80 parts of water and 5 parts of carbon black to form a thin paste, adding 7 to 9 parts of gasoline, agitating until the carbon black forms in pellets in the gasoline and leaves the water substantially clear, separating the pellets and the water, and drying the pellets.

24/17

Sworn at New York, in the County of New York and State of New York, this 30 day of November 1927.

William B. Wiegand
Louis J. Veruto

OATH

STATE OF NEW YORK :
: ss.
COUNTY OF NEW YORK :

WILLIAM B. WIEGAND and LOUIS J. VERUTO, the above-named petitioners, being duly sworn, depose and say that the former is a citizen of Canada and resident of Sound Beach in the County of Fairfield and State of Connecticut, and the latter is a citizen of the United States and resident of New York in the County of Queens and State of New York, and that they verily believe themselves to be the original, first and joint inventors of the improvements in CARBON BLACK AND PROCESS OF MAKING THE SAME described and claimed in the annexed specification; that they do not know and do not believe that the same was ever known or used before their invention or discovery thereof, or patented or described in any printed publication in any country before their invention or discovery thereof; or more than two years prior to this application, or in public use or on sale in the United States for more than two years prior to this application; that said invention has not been patented in any country foreign to the United States on an application filed by them or their legal representatives or assigns more than twelve months prior to this application; and that no application for patent on said improvements has been filed by them or their representatives or assigns in any country foreign to the United States.

William B. Wiegand
Louis J. Veruto

Subscribed and sworn to before me this 30 day of November 1927.

George W. Buck
NOTARY PUBLIC [illegible] No. 1977
Cert. filed in N. Y. Co. [illegible] No. 6458
Cert. filed in Kings Co. [illegible] No. 8479
Commission expires March [illegible] 1928
24/18

50 Room 322 DEPARTMENT OF COMMERCE Paper No. 2
XV/R UNITED STATES PATENT OFFICE
WASHINGTON

MAILED
MAY 29 1928

Please find below a communication from the EXAMINER in charge of this application.

Thomas E. Robertson
Commissioner of Patents.

Dean, Fairbank, Obright & Hirsch,
Graybar Bldg.,
New York, N. Y.

Applicant: W. B. Wiegand et al,
Ser. No. 237,230
Filed Dec. 2, 1927
For Carbon Black etc.

References made of record:

Krause,	1,198,039	Sept.12, 1916	210-208
Geer,	1,245,700	Nov. 6, 1917	106-23
Lewis,	1,2o3,o82	Apr. 16, 1918	106-23x
Randall,	1,405,439	Feb. 7, 1922	106-23
Spear,	1,511,984	Oct. 14, 1924	18-55
Spear,	1,560,488	Nov. 3, 1925	"

Claims 1-10 are rejected for lack of invention over Lewis and Randall above both of which disclose a substantially similar product.

Claims 11, 15, 18 and 19 are rejected as reading on Krause which mixes a water-oil mixture through a flaky carbonaceous material; the drying of the filter material for re-use or for removing the oil would be an obvious step if the material is to be used as a filler.

Claim 16 is rejected as broader than the disclosure; while applicants have disclosed other materials which could be treated like carbon black, they have not described the preferential agents which when used as in the described treatment of carbon black, would act likewise. The problem of discovering the proper combination of immiscible liquids which would act preferentially to wet the particles is a difficult and tedious problem requiring long and painstaking investigations. Applicants, having failed to disclose the combination of liquids suitable to carry on the process with the other materials, are not entitled to generically claim the treating of any material with any two immiscible liquids; In re Ellis 167 O.G. 981; In re Dosselmann & Heijkamp 167 O.G. 985.

Claim 22 is rejected as substantially met by either of Spear patents, both of which disclose the incorporating of vulcanizing agents or accelerators in carbon black.

Claims 1 and 2 are not patentably distinct.

Claim 5 is not patentably different from claim 3; the claims recite substantially the same product with a different phraseology.

Claim 7 is not patentably different from claim 4; the difference is in expressing the same thing in a different form; the content is the same.

Claims 12, 13, 14, 17 and 20-24 appear allowable.

3.m.

C. L. Blake

Examiner.

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3/A

MAIL DIVISION JUL 29 1928 U.S. PATENT OFFICE

IN THE UNITED STATES PATENT OFFICE.

Div. 50, Room 322,

Application of

WILLIAM B. WIEGAND and
LOUIS J. VENUTO

CARBON BLACK AND
PROCESS OF MAKING
SAME

Serial No. 227,330,

Filed Dec. 2, 1927.

U. S. PATENT OFFICE
FILED
JUL 30 1928
DIVISION 50

July 26, 1928.

Hon. Commissioner of Patents,
Washington, D. C.

Sir:

In response to the official action of May 29, 1928, we hereb amend the above entitled application as follows:

Page 11, after line 7, insert:

We use the term "carbon black" in its broad rather than limited meaning, and by such term include lamp black, gas black and other black analogous carbon substances for use as pigments or fillers.

After line 17, insert:

We have obtained very satisfactory pellets from magnesium carbonate and also from zinc oxide by use of the process exactly as described above although benzol has been used instead of gasoline.

Cancel claims 1, 2, 5, 7, 9, 10, 11 and 22.

Add the following claims:

~~25. (To be renumbered as claim 1)~~

1. Substantially pure carbon black in the form of commercially uniform, comparatively small, rounded, smooth aggregates having a spongy or porous interior.

26. The process of removing occluded gas from a light mass of carbon black particles and causing the particles to cohere in aggregates of greater density, which includes agitating it in the presence of two substantially immiscible liquids of different wetting power, and thereafter removing both liquids.

REMARKS

In view of the Examiner's ruling that certain of the claims are not patentably different from others applicants have reduced the number of claims and have presented new claims in place of the cancelled ones.

Reconsideration of the rejection of claims 3, 4, 6 and 8 is requested. These claims are not anticipated by either Lewis or Randall.

Lewis removes the air by the use of a wetting liquid which is then removed by evaporation.

One important object of applicants' invention is to avoid the harmful results which always follow such evaporation of water from carbon black and analogous materials.

Applicants' researches were directed to various ways and means of removing a wetting liquid from a pigment in such a way as to avoid the following objections:

a. A free meniscus. When a liquid containing a suspension of particles contracts in volume due to evaporation, the meniscus or free surface causes the particles to adhere just like two straws which run to each other when placed in a bowl of water. The Examiner can very easily verify this in his own kitchen sink. As a matter of fact there are various ways of avoiding such a meniscus, but the preferred manner mentioned in our application achieves this by means of displacing the wetting liquid by means of another liquid, employing the principle of preferential wetting.

b. Vortex action. When the liquid evaporates there is a violent small-scale vortex action which causes the suspended particles to adhere, so that when evaporation is complete, there results a cake which is of course undesirable.

c. Volume concentration. When the liquid is evaporated, it contracts in volume thus obviously compacting the suspended particles, and thus again tending to bring about a cake. By our method there is no concentration in the liquid which has displaced the air. There is a physical transfer to another liquid, thus avoiding concentration.

d. Variability due to sedimentation. By the Lewis method, the coarse particles sink more rapidly than the fine particles, so that as evaporation proceeds there is not an even and uniform cake formed, but a continual gradation of fineness with the coarse layers on the bottom and the fine layers on the top. By our method where there is no evaporation and therefore no lamination, the particles are maintained exactly in statu quo, uniformly distributed as regards fineness throughout the whole.

All of the foregoing objections are present when a wetting liquid is removed by evaporation as in the Lewis process, and not present in applicants' process.

One of the applicants in the present case is particularly acquainted with the process of the Randall patent because he was employed by the same company as Randall at the time that Randall developed his idea.

The plain fact is that the Randall patent was never successfully employed, the reason being that carbon black cannot be made to cohere by treating it with tar unless quantities of tar are employed as a binder almost equal in weight to the black itself. In commercial rubber compounding, carbon black is largely employed to correct exqessive softness in rubber compounds, in other words, to make them stiffer. The result is that if tar is included in the mixing in such amount as is necessary by the Randall process, the effects of the carbon black compounding are entirely lost since as is quite obvious the tar which is one of the most powerful softening agents used in the rubber business, breaks down and weakens the rubber compound even more than the carbon black builds it up.

As proof of the foregoing, we draw attention to the wellknown practice in the art of using only a small percentage of tar in rubber compounds, and nothing like the amount indicated in the Randall patent.

These defects caused the rejection of the Randall process, and the company for which he worked out the patent has never adopted this process. 24/35

More broadly, the whole aim and object of our invention was and is to provide a commercially available form of carbon and lamp black which shall nevertheless be essentially pure. Binders have been used from the earliest times to make carbon and other pigments more compact. For instance, thousands of years, B. C., the Chinese in their stick inks used glue and other such binding materials in conjunction with carbon black to make the black non-flying and compact and free from air. There is thus no essential novelty in employing a binder to make coherent masses of this and other pigments, and the whole trick and novelty of our patent is to obtain this end without the use of a binder. Such to our knowledge has not as yet ever been accomplished.

The carbon black of Randall is not the same as that of applicants. It has beenput into a similar form because the proportion of binder needed to attain cohering aggregates is so large that it is no longer right to describe the material as carbon black. It could be correctly described only as a combination in approximately equal amounts of carbon black and tar or other binding material in the form of adherent aggregates. This is entirely different from our product which is essentiall, the pure substance.

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The product defined in new claim 25 and in original claims 3, 4, 6 and 8 is not the product of the Lewis patent and cannot be made by the process disclosed in Lewis nor is it the product of the Randall patent or the result of the Randall process.

The claims are thought to be clearly patentable over these references.

The use of the term "cohering" in claim 8 implies the absence of such binding materials as used in Randall.

Claim 3 defines over Randall by calling for substantially pure carbon black.

Claim 4 defines a hardness which is not characteristic of Randall's pellets.

Claims 15, 18 and 19 are thought to be clearly patentable over Krause. The important step included in claims 15 and 18 is the agitation of the carbon black with the two immiscible liquids. There is no agitation in the Krause process. The brittle, porous, granular, flaky, carbonaceous particles, together with the ash, merely serve as a filter. Furthermore, the Krause patent does not bring the carbonaceous material into pellet form, as defined in claim 18, and the proportions in Krause are not such as are called for in claim 19. This claim specifically limits the proportions to such as will prevent the forming of a free meniscus.

Claim 11 has been rewritten to render it somewhat more definite. Ennese does not disclose any process for causing carbon black particles to cohere in aggregates of great density.

It is thought that the rejection of claim 16 is not proper. The same liquids which are disclosed in the application for the treatment of carbon black may be employed for the treatment of other fine powders, such as those mentioned in the third paragraph of page 11 of the specification. The two decisions referred to by the Examiner are therefore not pertinent to the case.

Under separate cover a package is being sent to the Examiner which includes samples of the product made by applicants process as well as a sample of that made by the Lewis process.

All of the claims are thought to be clearly patentable, and an allowance is requested.

RESPECTFULLY SUBMITTED,

WILLIAM B. WIEGAND and
LOUIS J. MENUTO.

Per Dean Fambrough Albright & Hirsch
Attorneys for Applicant.

[redacted]

E. Robertson
Commissioner of Patents.

Applicant: W.B.Wiegand et al,

Ser. No. 237,330
Filed Dec. 2, 1927
For Carbon Black etc.

Dean, Fairbank, Obright & Hirsch,
Graybar Bldg.,
New York, N. Y.

In response to communication filed July 28, 1928.

Additional reference made of record:

Evans, 1,175,732 Mar. 14, 1916 134-60

The above patent discloses a process of removing the free pure carbon from coal tar pitch by treating the latter with an aqueous alkali; after this treatment the pitch is removed by treating the aqueous mass with naphtha and violently agitating to dissolve the pitch which is removed in order to obtain a "perfectly pure carbon"; it should be noted that the purified carbon is heated to drive off any of the napntha left on the carbon due to its greater wetting power for it than the water contained in the pitch.

Claims 12, 13, 15, 16, 17, 18, 19 and 25 are rejected as not patentable over Evans for the reasons above.

Claims 4, 8 and 25 are rejected as not patentable over Lewis, Evans or Randall of record; the claims do not distinguish from the product of the patents, in that the limitations recited in these claims are more or less inherent in the patented products.

Claim 16 is again rejected as broader than the disclosure for the reason of record. Holland Glue Co., v Perkins Glue Co., 272 O.G. 817.

Claims 5, 6, 14, 20, 21 and 23 are allowed.

3.M.

C. H. Blake
Examiner.

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C-2166
45-JJ

JUL 18 29

IN THE UNITED STATES PATENT OFFICE

U. S. PATENT OFFICE
JUL 19 1929
DIVISION

In re application of :
WILLIAM B. WIEGAND and :
LOUIS J. VENUTO : CARBON BLACK AND PROCESS OF MAKING SAME
Filed: December 2, 1927 :
Serial No.: 237,330 :
Div. 50, Room 328 :

New York, N. Y., July 17, 1929.

Honorable Commissioner of Patents,
Washington, D. C.

S I R:

Responsive to the Official Action of January 21, 1929, please amend the above entitled application as follows:

Claim 4, line 1, before "soft" insert --substantially pure--

Cancel claim 19 without prejudice.

REMARKS

Reconsideration is respectfully requested.

The newly cited Evans patent has been carefully considered but its pertinency to the process or product of the present invention is not apparent. It does not relate to a process for treating carbon black or to a process in which carbon black exists as such in any portion of the operation. The starting material in the process of the Evans patent is a pitch containing finely divided hard carbon particles which are not comparable and cannot be made comparable by the process of the Evans patent to the material

known as carbon black or to the aggregates of soft carbon black particles produced by the process of the present invention. Furthermore, in the process of the Evans patent the carbon particles are initially present in a pitchy mass, the components of which, like the naphtha in which they are dissolved, have a greater wetting power than water, so that the addition of water to this pitchy mass does not perform the same function and is therefore not equivalent to the addition of water to the dry light bulky material known to the art as carbon black, to the treatment of which the present invention relates.

Claims 12, 13, 15, 17, 18 and 26 are directed to a process for treating carbon black, a material which contains a large amount of occluded gas. The Evans patent discloses no process in which any dry material containing occluded gases, comparable to carbon black, is treated in accordance with the process recited in these claims or for that matter by any other process, and there is nothing in the disclosure of the Evans patent to suggest that the advantageous results accruing from the improved process of the present invention as applied to such material could be produced by the procedure recited in the above noted claims. Reconsideration of the rejection of the above noted claims on the Evans patent is accordingly requested.

The rejection of claim 16 on the Evans patent should be withdrawn for the reason that it specifically recites wetting the finely divided substance to be treated with one liquid and agitating it with a second liquid immiscible with the first and having greater wetting properties. For the reasons pointed out above, it will be

PENNIE, DAVIS, MARVIN & EDMONDS

apparent that the Evans patent does not disclose the first step regardless of the fact that water may be present. Furthermore, the pitch itself cannot be treated as the liquid first referred to for the reason that the pitch and naphtha are not immiscible as required by claim 16.

The repetition of the rejection of claims 4, 8 and 25 on the Lewis and Randall patents of record and the further rejection of these claims on the newly cited Evans patent is not clearly understood. Claims 4 and 8 are specifically directed to a product comprising soft carbon black particles. Claim 25 is specifically directed to an article consisting of substantially pure carbon black. Claims 4 and 8 are specifically directed to a product of substantially uniform size while claim 4, in addition to being specific to an article comprising soft carbon black particles, defines the physical characteristics of the article as being readily broken down to a fine state of subdivision upon the application of slight pressure. The Evans patent as previously pointed out, regardless of the fact that it may relate to a process in which substantially pure carbon in a finely divided state may be produced, does not disclose a product composed of the characteristic soft carbon particles found in a product known to the art as carbon black. This is indicated by the fact that the hard cake formed by the process of the Evans patent must be ground to reduce it to a finely pulverized state. Furthermore, there is no disclosure in the Evans patent of a product composed of aggregates of uniform size.

The product of the Randall patent is not a substantially pure carbon black as defined by claims 4 and 25, nor is it substantially uniform as defined by claims 8 and 25. The Randall patent describes the product as a granular mixture consisting of small pellets ranging in size from material in powdered form to lumps the size of a walnut or larger.

The product produced by the process of the Lewis patent is identical to that produced by the process described in the second paragraph on page 2 of the specification of this application. This product consists of cakes or chunks of hard carbon particles. The cakes or chunks are not of uniform size and no longer contain the uniformly soft carbon particles characteristic of the product known to the art as carbon black. The product defined by claims 4, 8 and 25 are distinguished from this product for the reasons above pointed out and for the reasons recited in the specification of this application as originally filed. The process of the Lewis patent cannot produce a product such as that recited in claims 4, 8 and 25 so that it is not seen how the production of the improved product of the present invention can properly be held to be inherent in the process of the Lewis patent.

The rejection of claim 16 as broader than the disclosure is without authority under the decision in Holland Glue Co. vs. Perkins Glue Co. cited and cannot be sustained. The applicants herein describe a process applicable to carbon black and other fine powders, various examples of which were also recited. The applicants describe as one

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exemplification of the process of the invention wetting a fine powder with water and adding gasoline. This process, using these two liquids, is quite applicable to other powders such as those listed. It may be true that due to the undesirable odor sometimes left by gasoline, that in the treatment of certain fine powders for certain specific uses the use of other liquids is more desirable. If, for example, water and benzol alone were applicable to the treatment of other powders such as magnesium carbonate and zinc oxide, for example, and the liquids described as applied to carbon black were applicable to carbon black only in the treatment of that specific material, the problem of discovering the proper combination of immiscible liquids which would act satisfactorily with other materials might be considered as being left to the investigations of others. However, such is not the case in the present instance. The rejection of claim 16 as broader than the disclosure is accordingly untenable.

The process of the present invention appears to be clearly novel with the applicants herein and is based on the application of a broad principle which appears to be broadly novel with the applicants herein. The protection granted should accordingly not be restricted to a specific application of their novel process.

In the light of the above discussion, reconsideration and a favorable action are solicited.

Respectfully submitted,

Pennie, Davis, Marvin & Edmonds

Attorneys for Applicants.

2/1/34

PENNIE, DAVIS, MARVIN & EDMONDS

Div. 50 Room 322 Paper No. 355

Address only "The Commissioner of Patents, Washington, D. C." and not any official by name

XV/X

DEPARTMENT OF COMMERCE
UNITED STATES PATENT OFFICE
WASHINGTON

MAIL ED

JAN 4 1930

Please find below a communication from the EXAMINER in charge of this application.

Thomas E. Robertson
Commissioner of Patents.

Pennie, Davis, Marvin & Edmonds,
165 Broadway,
New York, N. Y.

Applicant: William B. Wiegand et al
Ser. No. 237,320
Filed Dec. 2, 1927
For Carbon Black and Process of Making Same

In response to Amendment filed July 18, 1929:-

Additional reference made of record:-

Acheson · 1,431,079 Oct. 3, 1922 134-1a

The above new cited patent makes a suspension of carbon black or other fillers in water and then intermixes a water-immiscible oil in the dispersion after which the carbon is recovered by addition of an electrolyte. Attention is called to page 1, lines 70 to 85, wherein it is stated that the flocculated mass has its bulk of water eliminated.

Claims 4, 8, 12, 13, 15, 16, 17, 18, 25 and 26 are rejected as lacking patentable novelty over Acheson above. It is evident that since the patentee treats the pigment in a manner similar to applicants' he will have a product substantially equivalent to applicants'

Rejection of Claims 4, 8, 25 as not patentably distinguishing from Lewis, Evans or Randall is repeated. Nearly all types of carbon blacks may be said to be soft, and when made from hydrocarbon oils there is obtained a substantially pure carbon particle, and of course when well dried, carbon blacks, even if slightly caked will crumble upon application of slight pressure.

Claim 16 is again rejected as broader than the disclosure for the reasons of record. "Substances in fine state of subdivision" include materials which may be inoperative, since they may react chemically with either wetting agent, or be dissolved by either or both.

Claims 3, 6, 14, 20, 21, 23, 24 are allowed.

O. F. Blade

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C 2166
45-AA

IN THE UNITED STATES PATENT OFFICE

In re application of,
WILLIAM B. WIEGAND, and
LOUIS J. VENUTO
Filed Dec. 2, 1927,
Serial No. 237,330,
Div. 50, Room 322

CARBON BLACK AND PROCESS OF MAKING SAME

JUL - 2 30

U. S. P. O.
JUL 3 1930
DIVISION 50

Honorable Commissioner of Patents,
Washington, D. C.

Sir:

Responsive to the Official Action of January 4, 1930, please amend the above entitled application as follows:

Claim 12, line 1, after "black" insert -- to produce a denser form of carbon black --.

Claim 13, line 1, after "black" insert -- to produce a denser form of carbon black --.

Claim 16, line 3, after "liquid" insert -- in which the finely divided substance is substantially insoluble and with which it does not react chemically --.

Line 4, after "second" insert -- similarly inactive --.

Claim 18, line 1, after "black" insert -- to produce a denser form of carbon black --.

PENNIE, DAVIS, MARVIN & EDMONDS

REMARKS

Reconsideration is respectfully requested.

Claim 16 has been amended so as to include only operations in which the substances in a fine state of subdivision are substantially insoluble in the liquids employed and in which the substances in a fine state of subdivision are substantially inactive chemically with respect to the liquids employed. It is quite obvious that such was the original intent of the claim. In view

of these amendments to claim 16, the rejection thereof as broader than the disclosure appears no longer pertinent and should be withdrawn.

The newly cited Acheson patent has been considered but its pertinency is not apparent. It is difficult to imagine two operations more foreign with respect to each other than the process of the Acheson patent and the process of the present invention. In the process of the Acheson patent the patentee emulsifies a water suspension of pigment with oil, whereas in the process of the present invention such emulsification is carefully avoided. Acheson co-precipitates oil and pigment by means of an electrolyte so that his final product includes oil plus pigment, whereas, in the process of the present invention the pigment is separated by means of the liquid and the final product is a pure pigment only. The purpose of the Acheson patent is to disperse pigments or powders in order to make a fluid coating composition and the patentee employs a vehicle which it would be difficult, if not impossible, to completely remove from the pigment, whereas the liquid of the greatest wetting power in the process of the present invention is a highly volatile liquid, all trace of which may be readily removed.

The Acheson patent does not disclose a process by which a compact and dustless but substantially pure product is obtained nor does it disclose a process by means of which such a product could be obtained. Furthermore, the Acheson patent does not suggest that the result which the applicants herein seek to accomplish by their process and do accomplish by such process, will be or can be accomplished by the process of the patent, by a similar process or by any process whatsoever. Certainly, the Acheson patent does not disclose an operation in which a product similar to that defined by Claims 4, 8 and 25 is formed as a final product or exists in any phase of the operation. The basis of the rejection of

PENNIE, DAVIS, MARVIN & EDMONDS

the products defined by claims 4,8 and 25 on the Acheson patent, we are therefore at a loss to understand. The Acheson patent does not disclose the formation of pellets of substantially pure carbon black particles as defined by claims 4 and 25 nor the formation of similar pellets of any other material. Similarly, the Acheson patent does not disclose a product consisting of carbon black particles or any other kind of particles cohering in small masses of substantially uniform size and having smooth outer surfaces as defined by claim 8. If the rejection of these three product claims on the Acheson patent is repeated, a further explanation of the rejection is requested, as we are unable to find in it a disclosure of a product either the same or similar to that defined by claims 4, 8 and 25.

The rejection of the process claims 15,16,17 and 26 on the Acheson patent is untenable. Each of the foregoing claims is directed to an operation involving not only treating carbon black or a similar substance in a fine state of subdivision with two liquids, but in addition requires the removal of both liquids from the final product. No such operation is disclosed in the Acheson patent, as the final product of the Acheson patent is a curd consisting of oil and pigment. Furthermore, in the process of the Acheson patent only a portion of the water is removed by the action of the electrolyte, as will be noted in lines 70 to 75 of page 1. In the process of the Acheson patent there is no suggestion of the formation of pellets nor is there the slightest suggestion as to what the nature of the final product would be if the oil could be and was removed from the curd formed in the Acheson patent by some undisclosed treatment. It will be noted that the mere removal of water in the process of the Acheson patent without the removal of the oil does not constitute the step of drying, as defined

PENNIE, DAVIS, MARVIN & EDMONDS

in claims 15 and 16, nor does it meet the requirement of removing the liquid of greatest wetting power as required by claims 17 and 26. The rejection of claims 15,16,17 and 26 on the Acheson patent should therefore be withdrawn.

Claims 12, 13 and 18 have been amended so as to define a process of treating carbon black to produce a denser form of carbon black. In their present form they are clearly distinguished from the disclosure of the Acheson patent which relates to a process in which the final product is an oil pigment card that is in no sense a carbon black of either light or dense form. Furthermore, the Acheson patent does not suggest that the operations defined by claims 12,13 and 18 would be of any utility in the treatment of carbon black to produce a denser form of carbon black. Reconsideration of these claims in their present form is accordingly requested.

The repetition of the rejection of claims 4, 8 and 25 on Lewis, Evans or Randall without further explanation is not understood. In the remarks accompanying the amendment of July 18, 1929, it was pointed out that the products defined by these claims were not disclosed in any of these three citations. The remarks set forth in the Office Action in connection with the repetition of this rejection throw no light on the basis of the rejection and are quite inconsistent with the definite teachings of the disclosures of these citations. For example, the Examiner states "Nearly all types of carbon blacks may be said to be soft," and adds "carbon blacks, even if slightly caked, will crumble upon application of slight pressure." However, the Examiner cites the Evans patent as an anticipation of claims 4, 8 and 25 which are specifically limited to a carbon black product, and the Evans patent definitely states that the caked product is of such a nature as to require grinding to obtain finely pulverized pure carbon.

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Obviously therefore, either the Examiner's statement to the effect that all types of carbon blacks will crumble upon the application of slight pressure must be an error or the Evans patent must relate to a form of carbon other than carbon black. In either case it will be clear for the foregoing reasons that the product of the Evans patent cannot be a product which will meet claims 4 or 25. Further, there is nothing in the disclosure of the Evans patent regarding uniformity of size of the cakes nor a smooth characteristic of the outer surface of the cakes as defined in claim 8, nor the shape or the characteristics of the surface or interior of the aggregates, as defined by claim 25. We are unable to find any description of these characteristics in any product disclosed in the Evans patent and it is respectfully requested, if the Examiner finds such a description in the Evans patent, that he point out more fully in what part of the patent such a disclosure may be found. On the other hand, if the rejection of these claims on the Evans patent is founded merely on the proposition that all forms of caked carbon black meet the requirements of these claims, attention is directed to the second paragraph of page 2 of the specification of this application describing several methods of treating carbon blacks resulting in the formation of hard cakes over which the process and product of the present invention are a distinct improvement. It is therefore submitted that the Evans patent should be withdrawn as a reference or its pertinency more fully explained.

Referring specifically to the Randall patent and the repetition of the rejection of claims 4, 8 and 25 on this reference, it may be conceded that the Randall patent discloses the formation of pellets, ranging in size from powdered form to lumps the size of a walnut. But certainly, such a disclosure is not a disclosure of a product consisting of

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particles of substantially uniform size as defined by claim 8. Furthermore, we are unable to find anything in the disclosure of the Randall patent regarding a pellet or a commercially uniform group of aggregates consisting of substantially pure carbon black. The product of the Randall patent, at least the only product we are able to find defined in the disclosure of this patent, is a mixture of tar and lamp black. If the Examiner finds a disclosure of a different product from the mixture of tar and carbon black above noted, in the Randall patent, it is respectfully requested that he more fully point out where in the Randall patent such disclosure may be found. The disconnected discussion concerning the fact that substantially pure carbon particles may be obtained from the hydrocarbon oils, appears to have no bearing on the pertinency of the Randall patent. The applicant herein is not claiming substantially pure carbon black as a new product, but it is quite obvious that the product of the Randall patent is not substantially pure carbon black in any form. The rejection of claims 4, 8 and 25 on the Randall patent should be withdrawn or some explanation given of the pertinency of this citation.

Referring specifically to the Lewis patent, attention is directed to the fact that nowhere in the specification is there any description of the character of the final product other than that it is more compact. There is nothing in the disclosure of this patent to indicate whether the final product is a large hard cake, a fine powder or a mixture of fine powder and large cakes. There is nothing to indicate the formation of rounded smooth aggregates having a spongy porous interior as defined by claim 25, nor the formation of a particle of substantially uniform size having smooth outer surfaces, as defined by claim 8. Attention is again directed to the fact that the

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process described in the first paragraph of page 2 of the Lewis patent is substantially the same as the operation defined in lines 20 to 25 of page 2 of the specification of this application, which has been found by the applicants herein to result in the formation of hard cubes which are in no way similar to the products defined by claims 4, 8 and 25. In view of the fact that there is nothing in the disclosure of the Lewis patent to indicate that the product formed by the process therein disclosed meets the requirements of the product defined by claims 4, 8 and 25 together with the fact that the applicants have found, as they have stated in their original specification, that the product formed by a process such as that of the Lewis patent is not a product similar to that defined by claims 4, 8 and 25 of this application, the basis of the rejection of these claims on the Lewis patent is not understood unless the Examiner finds in this patent something which a careful consideration has failed to disclose to us. We therefore, submit that the Lewis patent should be withdrawn as a reference or its pertinency more fully explained.

The Examiner in allowing certain of the claims in this application concedes the novelty both of applicants process and product and in the absence of any description in the Lewis, Evans & Randall patents of a product having the characteristics defined in claims 4, 8 and 25, we respectfully submit that the improved product of the present invention as defined in these claims is clearly patentable, and that in the absence of more pertinent art, these claims should be allowed. A careful reconsideration of all of the rejected claims in their present form, in the light of the above arguments, is respectfully solicited.

Respectfully submitted,

Bruce Barton, Barton & Brooks

Attorneys for Applicants

New York, N.Y.
July 1, 1930.

80 Form 332 DEPARTMENT OF COMMERCE Paper No. 9
UNITED STATES PATENT OFFICE
WASHINGTON

XV/K

FILED
DEC 19 1930

Please find below a communication from the EXAMINER in charge of this application.

Commissioner of Patents.

Pennie, Davis, Marvin & Edmonds,
165 Broadway,
New York, N. Y.

Applicant: William B. Wiegand et al
Ser. No. 237,320
Filed Dec. 2, 1927
For Carbon Black and Process of Making Same

In response to communication filed July 2, 1930:-

Rejection of Claims 4, 8, 12, 13, 15, 16, 17, 18, 26 as not patentable over the art and for the reasons of record is adhered to and repeated.

The Examiner has carefully considered the argument submitted by applicant but is still of the opinion that the above claims are unpatentable over the references. Rejection of the above claims is therefore made final.

Claims 3, 6, 14, 20, 21, 23, 24, 25 are allowed.

3. n.

C. L. Beake
Examiner.

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appeal

MAIL DIVISION JAN 15 31 U.S. PATENT OFFICE

U.S. Patent Office

IN THE UNITED STATES PATENT OFFICE

In re application of :

WILLIAM B. WIEGAND and
LOUIS J. VENUTO :

Filed December 3, 1927 : CARBON BLACK AND PROCESS FOR MAKING SAME

Serial No. 237,330 :

Div. 80, Room 332 :

OG NOISIAID
JUN 19 1931

Hon. Commissioner of Patents,
Washington, D. C.

Sir:

We hereby appeal from the final rejection, December 19, 1930, of Claims 4, 8, 12, 13, 15, 16, 17, 18 and 26 of the above entitled application "as not patentable over the art and for the reasons of record". We submit that the Examiner erred in rejecting the claims and that the Examiner erred in not allowing the claims. Our check for Fifteen Dollars ($15.00), the appeal fee, is enclosed herewith

Respectfully submitted,

Pennie, Davis, Marvin & Edmonds
Attorneys for Applicant

PENNIE, DAVIS, MARVIN & EDMONDS

New York, N. Y.,
June 12, 1931.

2743

JUN 16 1931 U.S. PATENT OFFICE

Enc.

DEPARTMENT OF COMMERCE
UNITED STATES PATENT OFFICE

MAILED
JUL 9 1931

In re Application of
William B. Wiegand and
Louis J. Vennito
Serial No. 297880
Filed December 2, 1927
For:- Carbon black and
process of making same

BEFORE THE BOARD OF APPEALS
ON APPEAL

EXAMINER'S STATEMENT

Appeal from the final rejection of claims 4, 8, 12, 13, 15, 16, 17, 18 and 20 which are as follows:

4. A pellet formed of substantially pure soft carbon black particles, the pellet being sufficiently hard to withstand ordinary shipment or handling, but readily breaking down to a fine state of subdivision upon the application of slight pressure.

8. Soft carbon black particles cohering in small masses of substantially uniform size and having smooth outer surfaces.

12. The process of treating carbon black to produce a denser form of carbon black which includes mixing it with one liquid, and adding a second liquid which is substantially immiscible with the first, and has greater wetting power for the carbon black.

13. The method of treating carbon black to produce a denser form of carbon black which includes the steps of mixing it with one liquid to remove the occluded gas, and separating the carbon black from said liquid by the action of a second liquid substantially immiscible with the first.

15. The method of treating carbon black which consists in agitating it in the presence of two immiscible liquids, whereby the carbon black forms pellets in or with one of said liquids, separating the pellets from the liquid, and drying.

16. The process of treating a substance in a fine state of subdivision to bring it to more compact form which consists in wetting it with one liquid in which the finely divided substance is substantially insoluble and with which it does not react chemically, agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the substance from the first liquid, and drying.

17. The method of treating carbon black which includes wetting it with one liquid, and then transferring it to a second liquid having greater wetting properties, and removing

the second liquid by evaporation.

18. The process of treating carbon black to produce a denser form of carbon black which includes agitating it in the presence of two substantially immiscible liquids of differing wetting power.

26. The process of removing occluded gas from a light mass of carbon black particles and causing the particles to cohere in aggregates of greater density, which includes agitating it in the presence of two substantially immiscible liquids of different wetting power, and thereafter removing both liquids.

The invention relates to a treatment of carbon black for increasing its density, removing any dust or gritty particles, reducing its oil absorption, and removing any occluded gas. The process consists broadly in submitting the carbon black to the simultaneous or successive treatment with two immiscible liquids, the second liquid having a greater wetting power for the carbon particles than the first liquid. More specifically, the carbon black is first mixed with water to form a thin paste, and then mixed with gasoline with agitation. The result of this process is that the carbon black particles absorb the gasoline and float on top of the water from which they are removed and dried to expell any absorbed gasoline.

The references relied upon are:

Evans	1,175,752	Mar. 14, 1916
Lewis	1,265,082	Apr. 16, 1918
Randall	1,403,439	Feb. 7, 1922
Acheson	1,481,076	Oct. 8, 1922

Evans discloses a process for obtaining pure carbon black from coal tar pitch by first treating the pitch with an aqueous alkali solution, and then violently agitating naphtha-water immiscible with the aqueous carbon-pitch mixture to dissolve the pitch and remove it to obtain a "perfectly pure carbon"; the purified carbon is heated to drive off any naphtha absorbed

by the carbon particles.

Lewis treats carbon black with liquids such as water, alcohol, gasoline, etc. by mixing the carbon black with the liquid and then drying to evaporate the liquid. It is stated that by this process there is obtained a carbon black of increased density (decreasing the volume causes increase in density) having any gas occluded removed, and whose softness is not impaired.

Randall treats carbon black with tar and naphtha to thoroughly saturate the black and then steam distil the naphtha leaving behind a dustless carbon black in the form of small pellets of increased density.

Acheson makes a suspension of zinc white, carbon black, etc., in water, and this suspension is mixed with a vehicle, such as linseed oil or a hydrocarbon oil of the paraffin type; the pigment-oil water mixture is now treated with an electrolyte and after stirring, allowed to settle into a curd-like deposit. These curds consist chiefly of oil and pigment from which the bulk of the water has been eliminated. This patent is silent as to the expelling of any hydrocarbon oil absorbed by the pigment, but since it is stated that, if any residual water is present, it may be evaporated off, such for example as passing the curd resins through steam heated rolls, it is evident that any hydrocarbon oil will be expelled during this treatment leaving a pigment of increased density, since compression will cause reduction in volume.

Claims 4, 8, 12, 13, 15, 16, 17, 18 and 26 are rejected for lack of invention over Acheson, while claims 4 and 8 are additionally rejected as not patentably distinguished from

Evans, Lewis or Randall and claim 16 is held broader than the disclosure.

The claims can be grouped into two groups:

(1) Claims 4, and 8 directed to a product; (2) claims 12, 13, 15, 16, 17, 18 and 26 directed to the process of treating the carbon black.

The process claims will be analyzed first; Claim 12 recites a process for producing a denser form of carbon black including mixing it with one liquid, and then adding a second liquid immiscible with the first and having a greater wetting power for the carbon black. Claim 13 is directed to the same subject matter with slightly modified terminology, but limited to the inherent function of the first liquid which "removes the occluded gas" from the carbon black. In claim 15 the carbon black is agitated in the presence of two immiscible liquids "whereby the carbon black forms pellets." Claim 16 is broadly directed to the treatment of a substance "in a fine state of division" similarly to claim 12. Claim 17 recites the wetting of the carbon black with one liquid, transferring it to a second liquid having greater wetting properties and removing the second liquid by evaporation (this claim implies that the first liquid may be left in the carbon black which is contrary to the teaching of the inventive concept). Claim 18 merely recites the treating of carbon black, to make it denser, including its agitating "in the presence of two substantially immiscible liquids of differing wetting power. Claim 26 is substantially of the same scope as claim 18 but claims "a process of removing occluded gas from carbon black particles, and causing the particles to cohere in aggregates of greater density.

From the above analysis it is seen that all the process claims, in one form or another, claim the treating of the carbon black particles with two immiscible liquids either simultaneously or consecutively, one liquid of which has a greater wetting power for the carbon black; several claims recite the functional purpose of the treatment which is to remove any occluded gas from the particles, and make the carbon black denser; these functions are the inherent and obvious result of the treatment.

Since Acheson first makes a water suspension of the carbon black and then agitates this suspension into a water immiscible oil which may be a vegetable or a paraffine oil, it cannot see in what respect applicant's process is patentably different from Acheson's; claim 17 recites exactly the same sequence of steps as Acheson's preferred form. The paraffine oil included the gasoline used by applimant. It may be argued that that Acheson states that he obtains an emulsion, while in applicants' process this is impossible; it is believed that when Acheson states that "to the resulting emulsion - - -" it is merely meant that a more or less homogeneous suspension of the carbon black in the two immiscible liquids is obtained; applicant must also obtain this type of dispersion until the first liquid is partially displaced by the second having a greater wetting power. That it must be so is evident from the statement made by Acheson that the flocculated pigment-curds "consist chiefly of oil and pigment from which the bulk of water has been eliminated." While it is true, Acheson states that he may use a little ammonia to emulsify the oil, this is not strictly necessary; besides, even if linseed oil is used, it is absolutely impossible to obtain a soap under the condition. The ammonia acting merely to give a more or less

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stable suspension of the pigment whether Acheson uses a paraffine oil or an oil of the paraffine series, both type are water immiscible and have a greater wetting power for the carbon black than the water.

It may perhaps be also argued that Acheson has not a product in the form of pellets and free from oil. It is not believed to be sustainable, while, it is true, Acheson flocculates deposit, it is evident that these flocculated particles may merely deposit on the surface of the water similarly to applicants', the acid disolving ordinarily in the water, leaving the pigment wetted with the oil. It is also true that Acheson's purpose is to prepare a pigment-oil material; this will be possible only when linseed oil is used, since if a hydrocarbon oil of the paraffine type is employed, this oil will be evaporated or more or less removed when the oil particles containing "any residual water" are passed through "steam heated rolls" (page 1, lines 78 and 79) this latter step will cause also an increase in the density of the pigment. Applicants evaporate the second liquid either at low or high temperature; in page 11, paragraph 1 they state that the pellets can be passed between rolls before evaporation of the solvent, and then "evaporate the gasoline"; it is believed that when Acheson evaporates any residual water from the pigment-oil mixture between steam heated rolls, the paraffine oil will be removed, since naphtha, kerosene, gasoline, etc. are oils of the paraffine series, and these oils have usually boiling point below the boiling point of water.

Particular attention is called to page 5, paragraph 2, of the specification in which the applicants state that they also

obtain the pigment-liquids mass "like an emulsion" which "flocculates into granules" the aim of Acheson.

Claims 4, and 8 directed to the product recite; claim 4 "a pellet of substantially pure soft carbon black particles --- sufficiently hard to withstand ordinary shipment and handling but readily breaking down to a fine state of subdivision upon --- slight pressure"; in claim 8 there is stated that the "soft carbon black particles cohere in small masses --- having smooth outer surfaces":

It is believed that Acheson has a product having these properties, since these properties are due to the particular process used, and it is evident that Acheson, which treats the carbon black particles in a way substantially similar to applicants; must have a product having substantially similar properties.

Claims 4 and 8 are additionally rejected as reading on the product of Evans, Lewis or Randall. Evans states that he has "an absolutely pure carbon; in finely divided state". Lewis purpose is to obtain a dustless carbon black of increased density in the form of flakes freed from any occluded gas. Randall describes his product as consisting of a granular mixture comprising "small pellets" of greater density, in aggregate form, but readily broken down in five subdivision by slight pressure.

Claim 16 is additionally rejected as broader than the disclosure for the reasons as fully set forth in office action of May 29, 1928, Paper No. 2, to which attention is respectfully called.

For the above reasons it is believed that claims 4, 8, 16,

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16, 16, 16, 17, 18 and 26 are not patentable over the references and their rejection should be sustained.

Respectfully submitted,

O. Rude

Examiner.

XY-ee

C-2166
67-WO

Brief

IN THE UNITED STATES PATENT OFFICE

In re application of)
WILLIAM B. WIEGAND & LOUIS J. VENUTO)
Serial No. 237,330) BEFORE THE BOARD OF APPEALS.
Filed December 2, 1927)
For: CARBON BLACK AND PROCESS OF MAKING SAME.)

B R I E F

This is an appeal from the action of the Primary Examiner in finally rejecting claims 4, 8, 12, 13, 15, 16, 17, 18 and 26 of the above noted application.

THE INVENTION

The invention described in this application and defined by the rejected claims is a carbon black product and the method of making it.

It should be noted at the start that carbon black is a specific product well known to the art and differing essentially in its properties and physical structure from lampblack, bone black, finely ground carbon and the like.

In Blacks and Pitches, by H. M. Langton (D. Van Nostrand Company, 1925, page 27) the author says:

> "Carbon black is the fluffy, velvety-black pigment produced in the form of an impalpable powder by the burning of natural gas against a metal surface. This black is not to be confused with lampblack, from which it differs in several respects, and which is made by entirely different methods. According to G. L. Cabot,[19] who is one of the pioneers of the American carbon black industry, certain printing-ink makers of New York and Philadelphia found that the soot, deposited by the suitable burning of artificial gas, gave a beautiful gloss and an intense tint to printer's ink, differing in both these respects from the older and more familiar lamp black."
>
> (19. - J.Soc.Chem.Ind., 1894, 13, 128.)

Carbon black is used in many industries and particularly in the manufacture of rubber, printer's ink, wax and resin compositions and the like. Incident to such use, it must be shipped and stored and peculiar difficulties have been encountered in this shipping and storing, on account of the great amount of space which a small amount of the material occupies.

All efforts to reduce the amount of space occupied by the carbon black have met with difficulty on account of the fact that the methods proposed not only reduce the space occupied, but change the structure of the carbon black so that it can not be readily dispersed when the time arrives to use it. Much of the beneficial effect obtained by the use of carbon black is obtained on account of its physical structure which permits it to be readily dispersed into an extremely fine state of subdivision. If, by any treatment, the carbon black becomes so agglomerated that it will not readily disperse into such a fine state of subdivision, its value is materially lessened.

Taking for an example carbon black used in rubber, it is necessary that the carbon black be added to the rubber and worked into the rubber so thoroughly that no agglomerations are left in the rubber undispersed. If the carbon black is put into the rubber in a hard, cake form, it is obvious that the dispersion can not be accomplished with little enough kneading of the rubber to be practical, and that if the kneading is carried on beyond certain limits, it is not only costly but damaging to the rubber itself.

In shipping or storing carbon black, however, it is highly desirable that the material occupy as small a space as possible, and also that it be in such a form that a

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cloud of the material will not be dispersed into the air every time it is subjected to the slightest agitation.

The present invention is an improved method for reducing the space occupied by carbon black, reducing the tendency of the carbon black to be scattered by a slight breeze or agitation and at the same time keeping the carbon black in a form from which it may be quickly and easily dispersed. This is done by forming pellets each comprising a small mass of carbon particles adhered together. The particles are strong enough to withstand ordinary handling and shipping but are fragile enough to be easily crushed into a powder between the fingers.

The process of this invention comprises dispersing the carbon black in a liquid such as water, adding a quantity of a different liquid immiscible with and having a greater wetting power than the first, agitating the mixture and allowing it to settle. The carbon black will be found to have accumulated in small pellets at the dividing line or interfere between the two liquids. It is removed and dried and small pellets of pure carbon black are thus formed.

The product is also claimed.

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THE REFERENCES

The main reference upon which the Examiner relies is a patent to Acheson. The process described by this patent differs from that of the applicant in several important particulars. Acheson proposes to prepare pigments including carbon black for shipping by compounding them with an oil such as linseed oil and does this by making an aqueous suspension of the carbon black and stirring this suspension into linseed oil or paraffin oil. The mixing

continues until an emulsion is formed and this emulsion is then flocculated with hydrochloric acid. The curds formed by this treatment are removed, the water driven off, and the curds comprising a mixture of linseed oil and carbon black are the final product.

The process and product in Acheson therefore differ essentially from the present invention because Acheson's process is not a process for making a denser form of carbon black but rather a process of making a mixture of carbon black and linseed oil. The product is not freed of linseed oil and hence is not pure, soft carbon black, but a mixture of carbon black and linseed oil. Furthermore, this product is not in the form of pellets but in the form of an oily mass not usable where dry carbon black is desired.

The patent to Randall is for a process of mixing lampblack and tar and the product is in the form of pellets of varying size of mixed tar and lampblack. The only two liquids used in this patent, if tar can be considered a liquid, are tar and naphtha, and these two liquids are readily miscible. The product, including tar, is not usable where dry carbon black is desired.

The Lewis patent describes the treatment of carbon black to increase its density, but this is done by the use of a single liquid. The carbon black is wetted with this liquid and dried. The product is not in the form of pellets, but is in hard cakes that can never be restored to the former soft condition even by vigorous grinding. This patent was known prior art and the process thereof is referred to in the specification.

The patent to Evans describes the production of finely divided carbon from pitch by dissolving away all of

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the other ingredients of the pitch. Carbon black is a distinct product and finely divided carbon is not at all similar. A weak solution of caustic is used for the first step in the dissolving process and this is followed by a treatment with naphtha. The naphtha and the caustic solution become mixed at one stage in the treatment, but before the carbon is completely separated from the other constituents of the pitch, the naphtha has been replaced several times and has washed the product completely free of the caustic solution. The final product is merely finely divided carbon which is obtained in a finely divided or hard caked condition and not in the form of firm but soft pellets. This patent is for a process of preparing finely divided carbon and not for a process of increasing the density of already prepared carbon black.

The references relied upon by the Examiner, having been considered, it is apparent that only one, namely Lewis, even attempted to make a denser form of pure carbon black and that Lewis used a different method and necessarily got a different result. It is also apparent that the present invention is really different in substance from the processes and products of the references and there remains only to point out that these differences are defined by the claims, to reply to the Examiner's objection to claim 16 and to direct a few remarks to the Examiner's statement.

PATENTABILITY OF CLAIMS

The product claims 4 and 8 were rejected upon all of the references. Acheson does not apply, because his product is not carbon black, but a mixture of carbon black and oil and is not in the form of small masses or pellets. Randall does not apply because his product is

not carbon black but a mixture of a pigment and pitch and the pellets which Rendall forms are not uniform in size, but vary "from a powder to the size of a walnut" (Page 1, lines 108 et seq.). This is important as it would prevent subsequent dispersion from taking place evenly. Evans does not apply because his product is finely divided carbon not carbon black, and it is not in the form of pellets. Lewis does not apply because his product is not soft pellets, but hard cakes. It is submitted that none of the references apply and these claims should be allowed.

Claims 12, 15, 15, 16, 17, 18 and 26 are rejected only upon Acheson.

Claims 12, 15, 18 and 26 all describe a process for treating carbon black to produce a denser form of carbon black, but Acheson does not do this. Acheson's product is a mixture of oil and a pigment which may be carbon black. The applicant's process is not one of forming a mixture and hence Acheson is not a proper reference. In addition, claim 26 definitely describes the removal of the second liquid and this is not done by Acheson.

Claim 15 describes the treatment of carbon black by agitating it in the presence of two immiscible liquids to form pellets and separating and drying the pellets. Acheson forms no pellets and does not dry his product by removing the oil as the applicant does his second liquid.

Claim 16 describes the treatment of substances in a finely divided state in general, but defines over Acheson by the inclusion of the drying step, that is, the removal of the second liquid, and this Acheson does not do.

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This claim is a process of treating a substance to bring it into a more compact form, and not a process of mixing it with another substance as is the Acheson process.

Claim 17 definitely describes the removal of the second liquid, thus differing essentially from Acheson.

OBJECTION TO CLAIM 16

Claim 16 has been rejected as broader than the invention because it is not limited to carbon black; but it is apparent that while the process described in this application is particularly applicable to carbon black, it may also be applied to other substances, as for example precipitated magnesium carbonate, zinc oxide and the like, which are mentioned on page 11 of the specification. The arbitrary limiting of the scope of this invention by the Examiner is obviously without foundation.

EXAMINER'S STATEMENT

It is not desired to criticize the Examiner's statement, but it is necessary to call attention to and correct certain erroneous statements and conclusions contained therein.

On page 2, lines 28 and 29, the Examiner states that Evans obtains "pure carbon black." This is incorrect. Evans obtains "finely divided carbon" which is totally different from carbon black in its physical structure, as is well known in the art.

On page 3, in referring to the Acheson patent, the Examiner assumes that since Acheson evaporates off excess water, he also evaporates off the oil and in this he is in error. The oil which Acheson prefers to use is linseed oil, and this oil is difficult to drive off, and is

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not driven off at the temperature at which water volatilizes. In fact, linseed oil polymerizes at elevated temperatures and it is impractical to attempt its removal by heat, and Acheson makes no attempt to do so. While Acheson mentions that he may use a hydrocarbon oil, it is probable that the hydrocarbon oil he contemplated was similar to linseed oil; that is, that it was a heavy oil, hard to volatilize. It is practically certain that the oil would not be driven off as the Examiner assumes. The inference drawn by the Examiner is obviously incorrect and even were it true, it is altogether too hazy to be a basis for a serious rejection.

On page 4, in analyzing the claims, the Examiner states that claim 17 implies that "the first liquid may be left in the carbon black." No separate treatment is necessary to remove the first liquid as the first liquid is removed from the carbon black by the treatment with the second, which has a greater wetting power.

On page 5, the Examiner argues about whether or not Acheson makes an emulsion. While this has not been urged as a distinguishing feature, it will be evident from reading Acheson's description, that he does make an emulsion and uses ammonia to stabilize it whereas the applicant is very careful not to make such an emulsion as it would prevent his pellets from forming.

Throughout the Examiner's statement, the Examiner speaks of hydrocarbon oils, as though they were gasoline, kerosene and the like; but the type of oils to which Acheson referred was undoubtedly the heavier paraffin oils similar in properties to linseed oil. The ordinary heavy lubricating oil is an example, and this is not at all easy to volatilize.

It is respectfully submitted that this Honorable Board should find that the Examiner erred in not allowing the claims appealed, and that said claims are properly allowable.

Respectfully submitted,

Penne Davis, Marvin & Edmonds

Attorneys for Applicants.

PENNIE, DAVIS, MARVIN & EDMONDS

380 Appeal No. 2743 Paper No. 8

Decision.

U. S. PATENT OFFICE
BOARD OF APPEALS
JAN 14 1932
MAILED

Appeal No. 2743

Hearing:
January 6, 1932

IN THE UNITED STATES PATENT OFFICE

BEFORE THE BOARD OF APPEALS

Ex parte William B. Wiegand and Louis J. Vennte.

Application for Patent filed December 2, 1927, Serial No. 237,330. Carbon Black and Process of Making Same.

Messrs. Pennie, Davis, Marvin & Edmonds for applicants.

This is an appeal from the action of the primary examiner finally rejecting claims 4, 8, 12, 13, 15 to 18 and 26.

Claims 4 and 16 are representative.

4. A pellet formed of substantilly pure soft carbon black particles, the pellet being sufficiently hard to withstand ordinary shipment or handling, but readily breaking down to a fine state of subdivision upon the application of slight pressure.

16. The process of treating a substance in a fine state of subdivision to bring it to more compact form which consists in wetting it with one liquid in which the finely divided substance is substantially insoluble and with which it does not react chemically, agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the substance from the first liquid, and drying.

The references relied on by the examiner are:

Evans et al,	1,175,732,	Mar. 14, 1916,
Lewis,	1,263,082,	Apr. 16, 1918,

Randall, 1,405,439, Feb. 7, 1922,
Acheson, 1,431,079, Oct. 3, 1922.

This application relates to condensing carbon black from its dry, light, powdery condition to relatively dense pellets. Allowed claim 14 rather broadly defines the process.

14. The process of treating carbon black which includes the steps of mixing the carbon black with a liquid to form a very thin paste, adding a second liquid in smaller amount and which is immiscible with the first liquid, agitating, separating the carbon black and second liquid from the first liquid, and then separating the carbon black and the second liquid.

The examiner has held that the appealed claims are not patentable over certain art and the patent to Lewis is the only one relied upon which is intended for condensing the dry powder to leave it dry and free of other material. This patent, however, employs a single liquid treatment rather than the two-liquid treatment of allowed claim 14.

The patent to Acheson leaves the product in a pasty mass and the patent to Randall binds the particles together by tar. The patent to Evans et al does not describe a method of condensing the dry powder but a manner of producing the powder originally. It is our opinion that none of the references anticipates appellants' manner of condensing the dry powder to a dry condition by the use of the two liquids having different wetting properties for the powder.

It is believed, however, that none of the appealed claims properly defines the invention disclosed in the application as filed. It is true that near the bottom of page 3 of the original specification is a statement that

the process includes the simultaneous or successive treatment with the two liquids but there is no description in the specification as to the manner in which the treatment may be carried out simultaneously. In the first paragraph of page 5 of the specification is a statement that the particles should be completely wetted with water before the gasoline is added. This would appear to indicate that the simultaneous treatment of the powder with the two liquids is not practical. In any event there is no disclosure of any way of carrying out the process by the simultaneous use of the liquids and we consider that appellants' process claims should be restricted to the successive treatment.

Claims 4 and 8 are broad article claims not in any way restricted to the method taught by appellants or even necessarily to the particular product produced by their process. We believe that a product as broadly stated as by these claims may result from methods far afield from that disclosed by appellants and not in any way suggested by them. These claims are so broad that the product might be quite different from appellants'. We do not consider, therefore, that claims 4 and 8 properly define appellants' invention.

The processes of claims 12 and 13 do not result in the production of the condensed dry pellets and we do not consider these claims properly define.

Claims 15, 16, 18 and 26 do not set forth the successive liquid treatment and for the reasons above stated we do not regard them as properly defining the in-

vention disclosed.

Claim 17 does not indicate that the second liquid is added to the first and therefore does not properly define appellants' method.

The decision of the examiner is affirmed in holding the subject matter of the appealed claims unpatentable for the reasons above stated.

J. M. Hoffman
Assistant Commissioner

M. J. Thurston
Examiner-in-Chief

E. J. Morgan
Examiner-in-Chief

BOARD OF APPEALS.

January 14, 1932

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FEB 29 1932

IN THE UNITED STATES PATENT OFFICE

In re application of

WILLIAM B. WIEGAND AND
LOUIS J. VENUTO

Serial No. 257,330

Filed December 2, 1927

For: CARBON BLACK AND PROCESS FOR MAKING SAME

BEFORE THE BOARD OF APPEALS

Washington, D. C.
February 26, 1932

Honorable Commissioner of Patents,

Washington, D. C.

Sir:

The Board of Appeals is respectfully requested to consider the following claim in view of its decision of January 14, 1932 and recommend the allowance of this claim to the Examiner in view of that decision:

27. The process of treating a substance in a fine state of subdivision to bring it to a more compact form which consists in wetting it with one liquid in which the finely divided substance is substantially insoluble and with which it does not react chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having great wetting properties, separating the substance from the first liquid and drying.

REMARKS

Certain claims in this application were finally rejected and appealed to the Board of Appeals. The final rejection by the Examiner was based upon certain references and an objection that one claim was broader than the invention. In deciding the case the Board of Appeals decided that the rejection of the Examiner was unsound and that the references did not apply, but found that the claims appealed did not properly define the invention in certain respects, and rejected the claims for this reason.

While the fact that the Board of Appeals advanced this new ground of rejection for the appealed claims would appear to give this applicant the right to have the application reopened for further prosecution, it is not the desire of this applicant to request such a reopening at this time.

What the applicant desires to do is to make a very small change in one of the rejected claims, namely claim 16, so that it properly defines the invention according to the decision of the Board.

The process described by claim 16 is a process for making pellets from a substance in a very fine state of subdivision and consists in wetting the substance first with one liquid and then with a second liquid which is immiscible with the first, agitating the two to form globules of the substance and the second liquid, separating the globules from the first liquid and drying the globules

3/1/67

to form pellets of the substance which was originally finely divided. In claim 18 as it was submitted to the Board of Appeals it was stated that the substance was treated with two liquids but it was not made clear whether the treatment with the two liquids was simultaneous or whether they were added successively to the material and the Board of Appeals held that for this reason the claim did not properly define the invention. By simply inserting the word "subsequently" at the proper place in the claim it is made clear that the treatment with the two liquids takes place successively and as this obviates the ground of rejection given by the Board of Appeals, Claim 18 is rewritten above to include this change and it is respectfully requested that this Honorable Board recommend the allowance of this claim, so amended, to the Examiner.

This request is made directly to the Board because the Examiner refuses to take the initiative in recommending the allowance of any claim.

Also, the applicant does not by the proposed amendment agree with the Board of Appeals that his process may only be carried out by <u>successive</u> treatment with two liquids as the object may in fact be accomplished by simultaneous treatment, but the amendment is proposed for the purpose of expediency.

Further, as it has been almost forty days since the original decision of the Board of Appeals, an immediate

22/68

decision upon this request would be greatly appreciated as it will save to the applicant his rights of appeal if necessary.

Respectfully submitted,

Pennie, Davis, Marvin & Edmonds

Attorneys for applicant.

388 Appeal No. 2743 Decision.

U. S. PATENT OFFICE
BOARD OF APPEALS
FEB 26 1932
MAILED

Appeal No. 2743

IN THE UNITED STATES PATENT OFFICE

BEFORE THE BOARD OF APPEALS

Ex parte William B. Wiegand and Louis J. Vemuto

Application for Patent filed December 2, 1927, Serial No. 237,330. Carbon Black and Process of Making Same.

Messrs. Pennie, Davis, Marvin & Edmonds for Applicants.

REQUEST FOR RECOMMENDATION OF MODIFIED CLAIM.

Appellants have modified one of the claims which were considered by us in the appeal decided January 14, 1932, in such a manner as to overcome the substantially new rejection which was introduced into the record.

For reasons stated in our decision we do not consider that the original claim was properly anticipated by the art relied upon, and since the amendment overcomes our rejection we regard this claim as allowable. In view of the fact that appellants are probably entitled to further consideration because of the new rejection made by us, we recommend the entry and allowance of the modified claim.

Assistant Commissioner
Examiner-in-Chief
Examiner-in-Chief

BOARD OF APPEALS.

February 26, 1932

Amendat (not entered)

APPLICATION
02166
FEB 29 32

IN THE UNITED STATES PATENT OFFICE

In re application of

Wiegand and Venuto

Carbon Black and Process for Making Same.

Filed Dec. 2, 1927

Serial No. 237,330

Div. 50.

U. S. PATENT OFFICE
FILED
FEB 20 1932
DIVISION 50

Do not enter

Feb. 27, 1932

The Commissioner of Patents,

Washington, D.C.

Sir:

In response to the decision of the Board of Appeals of Jan. 14, 1932, and the recommendation by the Board of Appeals dated Feb. 27, 1932, please amend the above entitled application as follows:

Cancel claims 4, 8, 12, 13, 15, 17, 18 and 26

Claim 16, line 3, before "agitating" insert

—simultaneously—

not in accordance with recommendation

REMARKS.

This amendment is made pursuant to the recommendation of the Board of Appeals and is believed to place the application in condition for immediate allowance. Such an allowance is respectfully requested.

Respectfully submitted,

Pennie, Davis, Marvin & Edmonds

Attorneys for APPLICANTS

PATENT APPLICATION DIV. MAR -1 32

IN THE UNITED STATES PATENT OFFICE

In re application of
Wiegand & Venture
Carbon Black and Process for Making Same
filed Dec. 2, 1927
Serial No. 237,330
Div. 50.

U. S. PATENT OFFICE
MAR 2 - 1932

Mar. 1st, 1932

The Commissioner of Patents,
Washington, D.C.

Sir:

In response to the decision of the Board of Appeals of Jan. 14, 1932, and their recommendation dated Feb. 27th, 1932, please amend the above entitled application as follows:

Cancel claims 4, 5, 12, 13, 15, 17, 18 and 26.

Claim 16, line 3, before "agitating" insert

—subsequently—

REMARKS.

As this amendment to claim 16 conforms to the recommendation made by the Board of Appeals, it is believed that the claim is now in condition for allowance and it is respectfully requested that the application be passed to issue in the near future.

Respectfully,

Parmelee Davis Watson & Edwards

Attorneys for APPLICANT.

Div. 50 Room 4701 Paper No. 19

Address only "The Commissioner of Patents, Washington, D. C." and not any official by name

XF/K

DEPARTMENT OF COMMERCE
UNITED STATES PATENT OFFICE
WASHINGTON

All communications regarding this application should give the serial number, date of filing, and name of the applicant

MAILED
MAR 14 1932

Please find below a communication from the EXAMINER in charge of this application.

Thomas E. Robertson
Commissioner of Patents.

Pennie, Davis, Marvin & Edmonds,
165 Broadway,
New York, N. Y.

Applicant: W. B. Wiegand et al

Ser. No. 237,330
Filed Dec. 2, 1927
For Carbon Black etc.,

In response to communications filed Feb. 29, 1932 and March 1, 1932:-

Added to the record:-

Spearman 1,377,937 May 10- 1921
Borcherdt 1,585,755 May 25- 1926

Spearman discloses the treating of graphite with water and kerosene, removing the water and evaporating the kerosene. Apparently the graphite is treated with the water and oil simultaneously, but there would be no invention in feeding the oil from tank 6 after the graphite is mixed with water. Applicant states that his process "includes the simultaneous or successive treatment" of the powdered substances with the two immiscible liquids.

Borcherdt treats powdered inert material by first making a water dispersion of the same, adding a selective agent including petroleum oils, removing the water, and releasing the selective oil by heat; see page 4, lines 114 to 130; page 5, lines 4 to 6; page 6, lines 14 to 20.

Claim 16 is rejected as not patentable over Spearman or Borcherdt for the explanation given above; Borcherdt reads directly on the claim.

Claims 3, 6, 14, 20, 21, 23, 24, 25 stand allowed.

C. H. Blake
Examiner.

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20/2

WASH. ROOM
SEP 12 1932
U.S. PATENT OFFICE

0 2166

IN THE UNITED STATES PATENT OFFICE

In re application of

WILLIAM B. WIEGAND AND
LOUIS J. VENUTO

Serial No. 237,330 — Div. 50.

Filed December 2, 1927,

For: CARBON BLACK AND
PROCESS FOR MAKING SAME.

Washington, D.C. Sept. 12, 1932.

U. S. PATENT OFFICE
FILED
SEP 12 1932
DIVISION 50

Hon. Commissioner of Patents,
Washington, D. C.

Sir:

Responsive to the Office action of March 14, 1932, please amend the above entitled application as follows:

Rewrite claim 16 as follows:

~~9. The process of treating compounding materials for use in plastic and coating compositions in a fine state of subdivision to bring it to more compact form which consists~~ 16. The process of treating compounding material which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which it does not react chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the ~~material~~ substance from the first liquid, and drying to obtain the material in a more compact form

Add cl. 16

REMARKS.

Claim 16 has been rewritten in order to bring out more clearly that the process is one for treating compounding

materials for use in plastic and coating compositions. In view of the fact that claim 16 has already been considerably amended, it is not thought advisable to make any further amendments to the claim.

While claim 16 has been rewritten as indicated above, it is not believed that the patents to Borcherdt and Spearman are pertinent to the claim as formerly worded, because they do not relate to the treating of a substance in a fine state of subdivision to bring it into a more compact form. Both of them relate to the treatment of ores and the mere fact that they employ the physical-chemical principle of selective wetting is of no moment, as that principle is old and well known, and applicant is making no claim to it per se. What applicant is claiming in claim 16, now rewritten as claim 27, is the application of this principle in a process of treating, as now stated, compounding materials for use in plastic and coating compositions, which are in a fine state of subdivision in order to bring them into a more compact form.

Whereas the problem confronted by applicant was one of bringing the finely divided particles into a more compact form, those which confronted Borcherdt and Spearman were ones of separation. Applicant is dealing with successive treatments with two homogeneous liquids. Borcherdt and Spearman, as well as other patentees in the ore flotation process which use the physical-chemical principle of selective wetting, require the treatment not with a homogeneous liquid, but with a mixture of liquids.

Referring specifically to the patents to Borcherdt and Spearman, it is to be noted that neither claim that the principle of selective wetting is new.

Borcherdt brings out no new application of this principle. He employs the well known existing means of flotation in combination with certain other steps calculated to separate undesirable colloidal material from the valuable mineral in the ore. Merely to cite that part of Borcherdt's patent which deals with the well known principle of selective wetting as an anticipation of claim 16 is tantamount to citing the whole art of ore flotation or to a rejection based merely on the fact that the principle is known.

The patent to Spearman is no more relevant than is the patent to Borcherdt. Spearman was specifically concerned with separation of graphite in the gangue so that it could be removed in a high state of purity "although in a more finely divided condition" (Page 1, lines27 and 28). On the other hand, the problem of applicants was not to obtain the carbon black in a finer state of subdivision, but in a more compact form. It seems that the ends sought by Spearman and applicants are directly opposite and that there is nothing in the Spearman patent which is at all anticipatory of applicants' method. The only feature in common is that both refer to the well known and old principle of selective wetting of materials.

In view of the differences between applicants' invention and those of Borcherdt and Spearman, and particularly in view of the fact that applicants are now claiming a process for treating compounding materials for use in plastic and coating compositions in which the principle of selective wetting is utilized, it is believed that claim 27 should

be allowed, and the application passed to issue.

Respectfully submitted,

Attorneys for Applicant.

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Div. 50 4701

DEPARTMENT OF COMMERCE
UNITED STATES PATENT OFFICE
WASHINGTON

MAILED
SEP 28 1932

Please find below a communication from the EXAMINER in charge of this application.

Thomas E. Robertson
Commissioner of Patents.

Pennie, Davis, Marvin & Edmonds,
165 Broadway,
New York, N.Y.

Applicant: William B. Wiegand, et al.
Ser. No. 237330
Filed Dec. 2, 1927.
For Carbon Black and Process of Making Same.

In response to amendment filed September 12, 1932.

Claim 27 is rejected on the art and for the reasons of record.

Applicant's argument has been carefully considered; the Examiner, however, is still of the opinion that the above claim is unpatentable for the reasons as fully set forth in Office action of March 14, 1932.

The rejection of Claim 27 is therefore made final.

Claims 3,6,14,20,21,23,24, and 25 stand allowed.

E.M.

O.L. Beake

Examiner.

9/12/32

APPLICATION DIV
SEP 26 32
C-2166

IN THE UNITED STATES PATENT OFFICE

In re application of
Wiegand & Venuto
Carbon Black and Process for Making Same
Filed Dec. 2, 1927
Serial No. 237,330
Div. 50.

Sept. 23th, 1932

SUPPLEMENTAL AMENDMENT

The Commissioner of Patents,
Washington, D.C.

Sir:

Supplementing the amendment filed on or about Sept. 12, 1932, please amend the above entitled application as follows:

Claim 27, line 1, after "materials" insert--in a fine state of subdivision to bring them into a more compact form for use in plastic and coating composition--

line 2, beginning with "for use" cancel through "form" in line 3.

last line, cancel " substance" and insert--material--

last line, after "drying" insert--to obtain the material in a more compact form--

Insert the following new claim:

28. The process of treating carbon black, which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which

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it does not react chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the carbon black from the first liquid and drying to obtain the carbon black in more compact form.

REMARKS.

This amendment is made after an oral interview with the Primary Examiner at which claim 27 was considered in connection with the patents to Spearman and Borcherdt.

At the interview it was agreed that the preamble to claim 27 would better express applicant's invention if it were worded to more clearly bring out that the process is one of treating compounding materials in a fine state of subdivision to bring them into a more compact form. It was the Primary Examiner's opinion that, as now worded, the preamble to this claim infers too much relative to the use of the material in a fine state of subdivision in plastic and coating compositions, and does not sufficiently stress the point that the process is one for bringing the material into a more compact form. The amendment to the last line of the claim is made so as to bring out that after drying the material is obtained in a more compact form.

It is believed that the amendments to claim 27 should clearly dispose of the patents to Spearman and Borcherdt as references. It was the Primary Examiner's opinion that, as formerly worded, claim 27 was anticipated by the patent to Spearman in which finely divided particles of graphite are agglomerated onto the flakes of graphite. While that is true, that product is only an evanescent one in the Spearman process, as when the graphite is dried, it assumes its discrete association. It should be borne

in mind, as pointed out in the remarks accompanying the amendment last filed, that the object of Spearman and also Borchardt is directly opposite that of applicant, as in Spearman and Borchardt the problem is not one of obtaining a finely divided material in a more compact form, but to break down the material into a more finely divided condition.

New claim 38 is presented herewith to cover a process similar to that set forth in claim 37, which is specific to carbon black. This claim patentably differentiates from the other claims in the application, for example claims 14 and 30, and is thought to be patentable over Spearman and Borchardt for the same reasons as have been pointed out in connection with claim 37. It is also thought that new claim 38 is patentable over Spearman and Borchardt for the further reason that the physical properties of carbon black are entirely different from the graphite of Spearman and the materials treated by Borchardt, and that it would be entirely unobvious to a person skilled in the art that the principle of selective wetting which Spearman and Borchardt use in obtaining their materials in a finer state of subdivision, could be utilized in a process in which carbon black is brought into a more compact form.

For the reasons set forth above it is respectfully submitted that claim 37 as amended and claim 38 are unquestionably patentable over the prior art, and should be allowed along with the other claims of the application.

Respectfully,

Attorneys for Applicants

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Paper 23

C2188

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MAIL DIVISION
NOV 4 32

IN THE UNITED STATES PATENT OFFICE

U. S. PATENT OFFICE
NOV 5 - 1932

In re application of :

WILLIAM B. WIEGAND and LOUIS J. VENUTO :

Serial Number 237,330 :

Filed December 2, 1927 :

Div. 50 - Room 4701 :

CARBON BLACK AND PROCESS OF MAKING SAME

Pat 1889429 No 29

Supplemental Oath

U. S. PATENT OFFICE
FILED
NOV 5 1932
DIVISION 50

STATE OF NEW YORK :
:ss.
COUNTY OF NEW YORK :

WILLIAM B. WIEGAND and LOUIS J. VENUTO, being duly sworn, depose and say: That they are the joint applicants in the above-entitled application. That the subject matter of the amendments filed September 13, 1932 and September 28, 1932 in the above-entitled application, and particularly the following claims, -

> 27. The process of treating compounding materials in a fine state of subdivision to bring them into a more compact form for use in plastic and coating composition which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which it does not react chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the material from the first liquid, and drying, to obtain the material in a more compact form.
>
> 28. The process of treating carbon black, which consists in wetting it with one liquid in which the finely divided material is substantially insoluble and with which it does not coact chemically, subsequently agitating it with a second similarly inactive liquid immiscible with the first and having greater wetting properties, separating the carbon black from the first liquid and drying to obtain the carbon black in more compact form. -

was part of their invention, was invented before they filed the above-entitled application, was not known or used before their invention, was not patented or described in a printed publication in any country more than two years before they filed the above-entitled application, was not patented in any foreign country on an application filed by themselves or

PENNIE, DAVIS, MARTIN & EDMONDS

their legal representatives or assigns more than twelve months prior to the above-entitled application, was not in public use or on sale in this country for more than two years before they filed the above-entitled application, and has not been abandoned.

William B Benjamin L.S.

Louis J Kuntz L.S.

PENNIE, DAVIS, MARVIN & EDMONDS

Subscribed and sworn to
before me this 2
day of November, 1932.

N Myers
Notary Public.

New York, New York,
November 3, 1932.

24/83

-2-

Patent specification is omitted here, as it appears as part of PLAINTIFF'S EXHIBIT 1.

IN THE DISTRICT COURT OF THE UNITED STATES FOR THE SOUTHERN DISTRICT OF WEST VIRGINIA

Civil Action No. 58 On U. S. Letters Patent No. 1,889,429

BINNEY & SMITH COMPANY, Plaintiff,

vs.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants

AMENDED COMPLAINT—Filed October 13, 1939

Now comes the plaintiff, Binney & Smith Company, a corporation of New Jersey, having a place of business at New York, New York, and for its complaint against the defendants United Carbon Company and United Carbon Company, Inc., avers:

1. This action arises under the patent laws of the United States, and jurisdiction is founded on U. S. Code, Title 28, Sec. 41 and Title 35, Sec. 70.

2. Defendant United Carbon Company is a corporation incorporated under the laws of the state of Delaware and has a regular and established place of business at Charleston, West Virginia, within the Southern District of West Virginia, where and elsewhere it has committed and threatened to commit the acts of infringement hereinafter alleged. Defendant United Carbon Company, Inc., is a corporation incorporated under the laws of the state of Maryland and has a regular and established place of business at Charleston, West Virginia, within the Southern District of West Virginia, where and elsewhere it has committed and threatened to commit the acts of infringement hereinafter alleged. Defendant United Carbon Company owns all of the capital stock of defendant United Carbon Company, Inc., and dominates and controls it.

3. On November 29, 1932, United States Letters Patent 1,889,429 were duly and legally issued to William B. Wiegand and Louis J. Venuto for "Carbon Black and Process of Making the Same" upon application duly filed December 2, 1927. On September 17, 1934, said William B. Wiegand and Louis J. Venuto assigned said Letters Patent to plain-

tiff by an assignment duly recorded in the United States Patent Office. Plaintiff has been, since September 17, 1934, and still is the sole owner of said Letters Patent and of any and all rights to recover for the infringement thereof.

4. Defendants, since the issue of said Letters Patent and the acquisition by plaintiff of its rights therein, have been and still are infringing the same by making, using and selling products which embody the patented invention, and will continue to do so unless enjoined by this Court.

5. Plaintiff has given due notice to defendants of said Letters Patent and their infringement thereof and has marked products sold by plaintiff, which embody the invention of the said patent, with the number of said Letters Patent, but notwithstanding, defendants have continued to infringe and to threaten to infringe said Letters Patent.

Wherefore plaintiff demands a permanent injunction against infringement of said Letters Patent by the defendants and those controlled by defendants, an accounting for profits and damages, and an assessment of costs against defendants and such other and further relief as to the Court may seem just.

Howard P. Klostermeyer, Price, Smith & Spilman, Attorneys for Plaintiff. Address: Kanawha Banking & Trust Building, Charleston, W. Virginia.

Of Counsel: Dean S. Edmonds, Pennie, Davis, Marvin & Edmonds, 165 Broadway, New York, New York.

JOINT AND SEVERAL ANSWER OF DEFENDANTS TO AMENDED COMPLAINT—Filed January 16, 1940

Civil Action No. 58

BINNEY & SMITH COMPANY, Plaintiff,

vs.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants

Now Come the defendants, United Carbon Company and United Carbon Company, Inc., and jointly and severally

reserving unto themselves all rights of exception to the deficiencies of the Amended Complaint filed in said cause, for their joint and several answer thereto respectfully show unto the Honorable Court the following:

1) Answering paragraph 1 of the Amended Complaint, defendants admit that the action purports to be an action arising under the patent laws of the United States and admit the jurisdiction of this Court.

2) Answering paragraph 2 of the Amended Complaint, defendants deny that they have, either jointly or severally, committed or threatened to commit any acts of infringement of plaintiff's patent within the Southern District of West Virginia or elsewhere. The other allegations contained in this paragraph are admitted.

3) Answering paragraph 3 of the Amended Complaint, defendants admit that United States Letters Patent No. 1,889,429 was issued on November 29, 1932 to William B. Wiegand and Louis J. Venuto but deny that the said Letters Patent was duly and legally issued to the said William B. Wiegand and Louis J. Venuto; defendants admit that the title to said patent was assigned to plaintiff by an instrument in writing dated September 17, 1934, but being without sufficient information, defendants neither admit nor deny plaintiff's present title to said Letters Patent and to its alleged right to recover for infringement thereof, and leave plaintiff to its proof.

4) Answering paragraph 4 of the Amended Complaint, defendants jointly and severally deny each and every allegation thereof.

5) Answering paragraph 5 of the Amended Complaint, defendants admit receiving what purports to be a written notice from plaintiff regarding defendants' alleged infringement of said Letters Patent, but jointly and severally deny that they have infringed, continue to infringe, or threaten to infringe the said Letters Patent. Defendants, being without sufficient knowledge as to the other averments contained in this paragraph of the Amended Complaint, neither admit nor deny the same, but leave plaintiff to its proofs.

6) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that Letters

Patent No. 1,889,429, and each and every claim thereof, is invalid and void because the alleged invention or discovery described and claimed therein, and all material and substantial parts thereof, had been, prior to the date of the alleged invention or discovery thereof by the alleged inventors named in said Letters Patent, or more than two years prior to the filing date of the application therefor, published, patented or described and contained in Letters Patent or other printed publications hereinafter set forth, and in other patents and printed publications, the dates, publishers, numbers and grantees of which defendants are now unable to supply and pray to be allowed to add hereto by amendment to this answer or otherwise.

United States Letters Patent.

207,013, Carter, Aug. 13, 1878.
377,901, Lawrence, Feb. 14, 1888.
671,804, Metcalf, Apr. 9, 1901.
1,175,732, Evans, Mar. 14, 1916.
1,191,673, Davis, July 18, 1916.
1,245,700, Geer, Nov. 6, 1917.
1,263,082, Lewis, Apr. 16, 1918.
1,285,363, Pike, Nov. 19, 1918.
1,286,024, Knowlton et al., Nov. 26, 1918.
1,377,937, Spearman, May 10, 1921.
1,405,439, Randall, Feb. 7, 1922.
1,431,079, Acheson, Oct. 3, 1922.
1,511,984, Spear, Oct. 14, 1924.
1,560,488, Spear, Nov. 3, 1925.
1,651,733, Sheppard et al., Dec. 6, 1927.
(Filed Nov. 26, 1926).

Foreign Patents.

Br. 11,684, Pringle, 1886.
Br. 127,770, Curbishley, 1919.
Br. 151,236, Trent.
Br. 183,430, Trent.
Br. 293,454, Trent, 1929.
(U. S. Convention date July 7, 1927.)

7) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that each and every one of the claims of said Letters Patent No. 1,889,429 is invalid and void in that the alleged invention

or improvement described and claimed therein was known and in public use in the United States before the alleged invention thereof by the alleged inventors named in said Letters Patent, or more than two years prior to the application for said Letters Patent, the names of such users and those having such knowledge and the places of certain of such public use being as set forth in the patents designated in the preceding paragraph.

8) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that each and every one of the claims of said Letters Patent No. 1,889,429 is invalid and void because the alleged invention purported to be patented thereby did not constitute patentable knowledge or invention within the meaning of the patent laws in view of the state of the prior art, including that set forth in the patents enumerated in said paragraph 6 hereof, and in view of what was common knowledge on the part of those skilled in the art, all prior to the time of the alleged invention by the alleged inventors named in said Letters Patent No. 1,889,429.

9) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that while the application for said Letters Patent No. 1,889,429 was pending in the United States Patent Office, the applicants therefor so limited and confined the claims of said application under the requirements of the Commissioner of Patents and because of the state of the prior art, that plaintiff is now estopped from obtaining any interpretation or construction thereof sufficiently broad to cover or include carbon black, carbon black products or any other product made, sold or used by defendants, or either of them.

10) Further answering said Amended Complaint, defendants and each of them aver and are ready to maintain and prove that plaintiff is without equity and is not lawfully or equitably entitled to the relief prayed for in the said Amended Complaint by reason of plaintiff's laches and delay in bringing said suit and filing said Amended Complaint.

11) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that each and every one of the claims of said Letters Patent No. 1,889,429 is invalid and void because the same do not set

forth, describe or claim any lawfully patentable invention, but on the contrary describe wholly unpatentable features of a carbon black pellet or a process for the manufacture thereof, all of which is within the knowledge of those skilled in the art to which the alleged invention pertains.

12) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that claims 1 to 3, inclusive, of the said Letters Patent No. 1,889,429, and each and every one of the said claims, is invalid and void as attempting to cover a product which is the result or function of the process disclosed in said patent and claimed in claims 4 to 10, inclusive, and as failing to set forth and particularly describe the alleged novel product in terms which define the scope of the said patent and distinguish the alleged novel product from products previously known in the art.

13) Further answering said Amended Complaint, defendants aver and are ready to maintain and prove that claims 1 to 3, inclusive, of the said Letters Patent No. 1,889,429, and each and every one of the said claims, is invalid and void as failing to make a disclosure sufficiently definite to satisfy the requirements of Sec. 4888 of the Revised Statutes, 36 U. S. C. A. Sec. 33.

Wherefore, defendants and each of them pray that the Amended Complaint filed in this cause be dismissed and that costs be awarded to defendants for and on account of their defense thereof.

United Carbon Company and United Carbon Company, Inc., by Osman E. Swartz, Their Attorney, Business Address: % United Carbon Company, Charleston, West Virginia. Dike, Calver & Gray, Arthur M. Smith, of Counsel, 2343 Union Guardian Bldg., Detroit, Michigan.

Dated: January 15th, 1940.

A copy of the foregoing Joint and Several Answer of Defendants to Amended Complaint has been served on Messrs. Price, Smith & Spilman, attorneys for the plaintiff herein, by mailing the copy of such document to them at Kanawha Banking & Trust Company Bldg., Charleston, West Virginia, on the 15th day of January, 1940.

Osman E. Swartz, Attorney for Defendants.

440

BILL OF PARTICULARS

(Filed December 18, 1939)

Civil Action No. 58

U. S. Letters Patent No. 1,889,429

BINNEY & SMITH COMPANY, Plaintiff,

vs.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants

Now comes the plaintiff, Binney & Smith Company, and, in response to defendants' motion for a more definite statement of claim or bill of particulars, states:

1. As at present advised, and with respect to that product of the defendants a sample of which has been supplied by defendants to plaintiff, plaintiff alleges that claims 1 and 2 of patent 1,889,429 are infringed (a) by making, (b) by using, and (c) by selling this product.

2. The particulars requested in this paragraph of the motion are regarded as unnecessary in view of the fact that defendants have supplied plaintiff with a typical sample of the product the manufacture, use and sale of which constituted infringement upon which this suit is based.

3. As at present advised, and with respect to the product a sample of which has been supplied by defendants to plaintiff, plaintiff does not rely upon any of claims 4 to 10 of said patent 1,889,429.

4. A photostatic copy of the instrument upon which plaintiff relies to establish its title to Letters Patent 1,889,429 is attached hereto and made a part hereof.

Binney & Smith Company, by Howard R. Klostermeyer, Price, Smith & Spilman, Attorneys for plaintiff. Dean S. Edmonds, of Counsel.

Service of within bill of particulars and attached assignment therein referred to is hereby accepted this 18th day of December, 1939.

Osman E. Swartz, Attorney for United Carbon Company and United Carbon Company, Inc.

ASSIGNMENT

Liber W160 Page 262.

Whereas, We, William B. Wiegand, a citizen of Canada and a resident of Sound Beach, County of Fairfield and State of Connecticut, and Louis J. Venuto, a citizen of the United States, and a resident of New York in the County of Queens and State of New York, have invented certain new and useful improvements in "Carbon Black and Process of Making Same" for which we filed an application for Letters Patent of the United States December 2, 1927, Serial No. 237,330, on which Letters Patent of the United States Number 1,889,429 were granted November 29, 1932, and

Whereas, Binney & Smith Co., a corporation organized and existing under the laws of the State of New Jersey and having a place of business at 41 East 42nd Street, New York, New York, desires to obtain the entire right, title and interest in, to and under the said improvements and the said Letters Patent:

Now, Therefore, in consideration of the sum of One Dollar ($1.00) to us in hand paid, and other good and valuable consideration, the receipt of which is hereby acknowledged, we, the said William B. Wiegand and Louis J. Venuto have sold, assigned, transferred and set over, and by these presents do hereby sell, assign, transfer and set over unto the said Binney & Smith Co., the entire right, title and interest in, to and under the said improvements, the said application and all divisions, continuations and renewals thereof, the said Letters Patent of the United States and all Letters Patent of the United States which may be granted on said application, divisions, continuations and renewals thereof, and all re-issues and extensions thereof, and all applications for Letters Patent for said improvements filed in any country or countries foreign to the United States and all Letters Patent granted for said improvements in any country or countries foreign to the United States and all extensions, reissues and renewals thereof, together with the right to recover for all past infringement of said Letters Patent of the United States; we hereby authorize and request the Commissioner of Patents of the United States, and any Official of any country or countries foreign to the United States whose duty it is to issue patents on applications as

aforesaid, to issue all Letters Patent for said improvements to the said Binney & Smith Co., its successors, legal representatives and assigns, in accordance with the terms of this instrument.

And We Hereby covenant that we have full right to convey the entire interest herein assigned, and that we have not executed, and will not execute, any instrument in conflict herewith.

And We Hereby further covenant and agree that we will communicate to the said Company, its successors, legal representatives and assigns, any facts known to us respecting said improvements, and testify in any legal proceeding, sign all lawful papers, execute all divisional, continuing and reissue applications, make all rightful oaths, and generally do everything possible to aid the said Company, its successors, legal representatives and assigns, to obtain and enforce proper patent protection for said improvements in all countries.

In Testimony Whereof, I hereunto set my hand and seal this 17th day of September, 1934.

(Signed) W. B. Wiegand. L. S.

STATE OF NEW YORK,
County of New York, ss:

On this 17 day of Sept., 1934, before me, a Notary Public in and for the State and County aforesaid, personally appeared William B. Wiegand, to me known and known to me to be the person of that name who signed and sealed the foregoing instrument, and he acknowledged the same to be his free act and deed.

(Signed) Henry C. Klein, Notary Public. Notary Public Queens Co. No. 954, Reg. No. 4747, Cert. filed in N. Y. Co., No. 61, Reg. No. 5-K-42. Commission expires March 30, 1935. (Notarial Seal.)

In Testimony Whereof, I hereunto set my hand and seal this 17th day of September, 1934.

(Signed) Louis Joseph Venuto. L. S.

STATE OF NEW YORK,
County of New York, ss:

On this 17 day of Sept., 1934, before me, a Notary Public in and for the State and County aforesaid, personally appeared Louis J. Venuto, to me known and known to me

to be the person of that name, who signed and sealed the foregoing instrument, and he acknowledge the same to be his free and and deed.

(Signed) Henry C. Klein, Notary Public. Notary Public Queens Co. No. 954, Reg. No. 4747, Cert. filed in N. Y. Co., No. 61, Reg. No. 5-K-42. Commission expires March 30, 1935. (Notarial Seal.)

Recorded: Transfers of Patents, U. S. Patent Office, Sep. 19, 1934. Liber W 160 Page 262. ——, Commissioner of Patents.

IN THE UNITED STATES DISTRICT COURT, SOUTHERN DISTRICT OF WEST VIRGINIA

Civil Action No. 58

BINNEY & SMITH COMPANY, Plaintiff,

vs.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants

Notice of Patents To Be Relied Upon as Showing State of the Art

(Filed December 6, 1940)

To Messrs. Price, Smith & Spilman, Attorneys for Plaintiff, Kanawha Banking & Trust Company Bldg., Charleston, West Virginia.

SIRS:

Please Take Notice that on the trial of the above cause we shall rely upon the following patents as showing the state of the art prior to December 2, 1927:

United States Letters Patent

598,646, Saunders, Feb. 8, 1898.
809,354, Bartlett et al., Jan. 9, 1906.
1,076,235, Rodman, Oct. 21, 1913.
1,106,087, du Pont et al., Aug. 4, 1914.
1,114,029, Payne, Oct. 20, 1914.

1,116,044, Ellis, Nov. 3, 1914.
1,184,397, Weinrich, May 23, 1916.
1,239,221, Rodman, Sept. 4, 1917.
1,239,368, du Pont, Sept. 4, 1917.
1,422,698, Grossmann, July 11, 1922.
1,466,571, Thorssell et al., Aug. 28, 1923.
1,478,730, Brownlee et al., Dec. 25, 1923.
1,496,314, Ikeda, June 3, 1924.
1,541,233, McMahan, June 9, 1925.
1,561,971, Coffin et al., Nov. 17, 1925.
1,570,086, Schaffer, Jan. 19, 1926.
1,756,896, Wisner, Apr. 29, 1930.
(Filed August 7, 1926.)
1,783,983, Runge, Dec. 9, 1930.
(Filed June 12, 1925.)

Foreign Letters Patent

(Br.) 244,461, Societe de Recherches et al., 1927.

Osman E. Swartz, Attorney for Defendants, Union Building, Charleston, West Virginia. Dike, Calver & Gray, of Counsel.

December 4", 1940.

Receipt of a copy of the foregoing Notice is acknowledged this 6 day of December, 1940.
Price, Smith & Spilman, Attorneys for Plaintiff.

DEFENDANTS' EXHIBIT NO. 32

TAMMS PAINT MIXER and Agitator



$17.50 MOTOR EXTRA

AC MOTOR
¼ hp $7.95

TURN YOUR OLD PAINT STOCK INTO LIQUID ASSETS

This Means Repeat Business for You and Converts Old Stock Into Active Profits!

Every TAMMS Paint Mixer and Agitator is thoroughly tested before it leaves the factory—and is guaranteed for a period of one year against defects in materials and craftsmanship. It completely mixes the paint, giving a smooth, uniform shade, ready for application. Mixing time required depends on grade, quality and age of paint stock.

SPECIFICATIONS

- Easily adjusted to fit pints, quarts, half gallons and gallon cans.
- Extra strong crank shaft for dependable long service.
- Including motor, the mixer occupies a space only 10 x 22 inches. Will operate on a ¼ HP motor. Will mix any kind of liquid—cans do not have to be opened.
- TAMMS Paint Mixer and Agitator comes complete with 2" pulley and V-belt (motor not included). Shipping weight 40 pounds f.o.b. Chicago. Complete instructions for installation included with each mixer.
- One one-quarter HP motor can be supplied at a cost of $7.95. This motor is designed for 110 volts, 60 cycles, alternating, 1725 RPM. Motor prices for other currents will be supplied on request. Specify current, also AC or DC.

TAMMS SILICA CO., 228 N. La Salle St., Chicago, Ill.

C. CARTER.

Coated Compressed Medicaments.

No. 207,013. Patented Aug. 13, 1878

Fig. 1.



Fig. 2.



Fig. 3.



Fig. 4.



Witnesses:

R. F. Brandt

W. C. Fischer

Inventor:

Chas. Carter,

by John A. Wiedersheim

ATTORNEY

UNITED STATES PATENT OFFICE.

CHARLES CARTER, OF PHILADELPHIA, PENNSYLVANIA.

IMPROVEMENT IN COATED COMPRESSED MEDICAMENTS.

Specification forming part of Letters Patent No. 207,013, dated August 13, 1878; application filed April 2, 1878.

To all whom it may concern:

Be it known that I, CHARLES CARTER, of the city and county of Philadelphia, and State of Pennsylvania, have invented a new and useful Improvement in Coated Compressed Medicaments, &c., which improvement is fully set forth in the following specification and accompanying drawings, in which—

Figure 1 is a side elevation. Fig. 2 is a plan view. Fig. 3 is a section in line *x x*, Fig. 1. Fig. 4 is a section in line *y y*, Fig. 1.

Similar letters of reference indicate corresponding parts in the several figures.

My invention consists in the hereinafter-described improvement in the manufacture of coated compressed medicaments, the same consisting in partially enveloping the medicaments in coverings of a saccharine nature in the form of powder, and then applying a top or finishing covering of powder, and compressing the whole, so that the powder covering holds by cohesion, thus producing a seamless coated compressed medicament, substantially as described.

In carrying out my invention, I take a quantity or dose of the medicament and completely envelope it in a covering of sugar or equivalent material, prepared in any desirable manner, and then subject the whole to pressure, substantially as hereinafter more fully described.

It will be seen that the inclosed substances are preserved so as not to be affected by time or changes of temperature, climate, and season, and, if they are unpleasant to the senses, they are disguised, and may be readily swallowed without nausea, and also without inconvenience, as the size of the complete article is reduced, the solubility of the same in the digestive organs being in nowise prevented.

In the manufacture of the compressed coated article heat is not necessarily employed, as the coating and the compression may be accomplished at ordinary temperature.

The inclosed substance may be in the form of liquid, powder, paste, mass, or solid, and the covering composed of sugar or other similar material, and in a powdered condition.

In furtherance of the above, a suitable quantity of sugar, in powder, at ordinary temperature, and of ordinary degree of moisture of the atmosphere, is placed in the bottom of a compressing apparatus. The substance to be coated, if of a solid condition, is then introduced upon the sugar, sufficient space being allowed between the substance and the wall of the apparatus, as well as the upper part thereof, to permit the admission of surrounding or top-closing sugar, which being applied, the compressing-force is exerted, so that the particles of sugar are made to cohere and become solid, whereby, when the article is removed from the apparatus, the inclosed substance is completely and densely covered.

When a substance in condition of liquid or powder is to be covered or coated by compression, it may become necessary to modify the process by combining with the sugar a mucilage, gum, or other harmless adhesive material. A depression is made in the coating material of size to insert the substance in the condition just stated, the upper part whereof is closed with some of the coating material, and the whole subjected to sufficient compression.

I am aware that it is not new to cover medicaments, confectionery, &c., with sugar, gelatine, and other bland materials, and that compressed and coated pills are not new; but I am not aware that any substance has been covered or coated by compression in a manner hereinbefore stated and with the results set forth.

Heat has been employed as a medium of coating, and the want of ready solubility of the product thereof is an objection to the use of the latter, which is overcome by my cold process, and, as several other advantages are presented, as has been stated, I believe that I have made an improvement in the art.

I am also aware that capsules have been before formed by gluing together concave disks of wafer, inside of which were placed proper medicaments; but in such case the disks formed the shell of the capsule.

In my invention a suitable mold or die, being coated with dry powdered sugar, or other similar material, on its inner surface, receives the medicament, and, compression being applied after a top coating or covering has been placed thereon, the sugar coheres to the compressed article.

What I claim as my invention is—

1. The herein-described improvement in the manufacture of coated compressed medicaments, the same consisting in partially enveloping the medicaments in coverings of a saccharine nature in the form of powder, and then applying a top or finishing covering of powder, and compressing the whole, so that the powder covering holds by cohesion, thus producing a seamless coated compressed medicament, substantially as described.

2. As a new article of manufacture, a compressed pill having a compressed seamless coating of powdered sugar or like substance, substantially as herein specified.

CHARLES CARTER.

Witnesses:

JOHN A. WIEDERSHEIM,

A. P. GRANT.

No. 671,804. Patented Apr. 9, 1901.

F. H. METCALF.

COATED PILL, &c.

(Application filed May 28, 1900.

(No Model.)

Fig. 1.

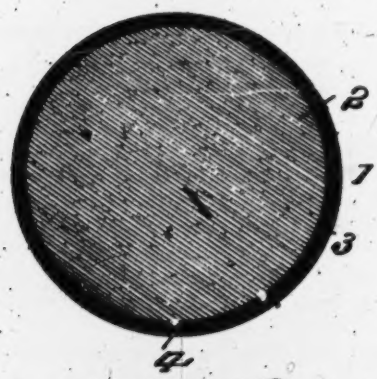
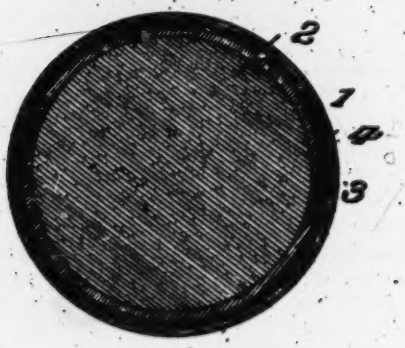


Fig. 2.



Witnesses
H. J. Hauptman
Wm. J. Jacobi

Inventor
Frederick H. Metcalf

By Jno. Imrie
his Attorney

UNITED STATES PATENT OFFICE.

FREDERICK H. METCALF, OF FRANKLIN, ILLINOIS.

COATED PILL, &c.

SPECIFICATION forming part of Letters Patent No. 671,804, dated April 9, 1901.

Original application filed December 2, 1899, Serial No. 738,993. Divided and this application filed May 28, 1900. Serial No. 18,207. (No model.)

To all whom it may concern:

Be it known that I, FREDERICK H. METCALF, a citizen of the United States, residing at Franklin, in the county of Morgan and State of Illinois, have invented new and useful Improvements in Coated Pills, Tablets, or Lozenges, of which the following is a specification.

This invention relates to improvements in coated pills, tablets, lozenges, and the like, and forms part of a divisional application filed December 2, 1899, Serial No. 738,993, allowed May 2, 1900, and patented under No. 650,760.

One object of this improvement is to mix with the coating used in the manufacture of coated pills a sufficient quantity of pepsin to quickly dissolve said coating and make the medicine which is incased more susceptible to the digestive organs when taken into the stomach.

A further object of the improvement is to mix a sufficient quantity of pepsin with the coating for pills when said coating consists primarily of gelatin so as to assist the stomach to quickly and readily digest the coating to permit the exposure of the incased medical compound to the digestive organs.

Many other objects and advantages will be apparent upon further description.

In the drawings forming a part of this specification, Figure 1 is a sectional view of a pill, showing my improvement applied thereto. Fig. 2 is a similar view showing a modified form of my invention.

Referring to the drawings, the numeral 1 represents a pill as a whole, composed of any suitable medical compound 2 and provided with a coating 3. Mixed with the coating is a sufficient quantity of pepsin or other digestive agent 4.

In the manufacture of pills, tablets, or lozenges for certain reasons it is necessary to provide a coating. In some cases this coating is composed of a mixture which is not readily susceptible to the digestive organs of the stomach; and it is the purpose and object of this invention to introduce in its composition a sufficient quantity of pepsin so as to enable the stomach to readily and quickly reduce the coating and expose the incased medical compound. The coatings for pills, as before stated, are largely made up of a gelatinous substance, and it is well known this substance is exceedingly difficult to digest, and it not infrequently occurs that a gelatin-coated pill absolutely refuses to be subjected to the digestive organs and juices of the stomach, and as a result the pill is taken without any curative result.

While I am aware that the coating of pills is largely gelatin, I am also aware of other substances being used equally as hard to digest and propose to mix pepsin with my coating solution used for pills.

The pepsin is applied to the pill, tablet, or lozenge preferably when the coating is in a liquid state. However, a separate compartment may be made, as disclosed in Fig. 2. In this modification the pepsin is contained between two layers of coating, the outer one of which is thinner than the inner, so much so as to not be a barrier to the juices.

In many cases I may apply enough pepsin to not only reduce the coating around the pill, but to coact with the compound incased within the coating and assist in its digestion.

Having thus described my invention, what I claim is—

1. As a new article of manufacture, a pill, tablet or lozenge provided with a gelatinous coating, said coating having pepsin distributed throughout its composition to assist the digestion of said coating and the rapid liberation of the compound forming the pill, whereby the juices of the stomach, may have immediate action on said compound, substantially as described.

2. As a new article of manufacture, a pill, tablet or lozenge, provided with a gelatinous coating having pepsin uniformly distributed throughout its composition to assist the digestion of said coating and the rapid liberation of the compound forming the pill, whereby the juices of the stomach may have immediate action on said compound, substantially as described.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

FREDERICK H. METCALF.

Witnesses:
CHARLES A. BARNES,
LENA C. ENGEL.

[This Drawing is a reproduction of the Original on a reduced scale]

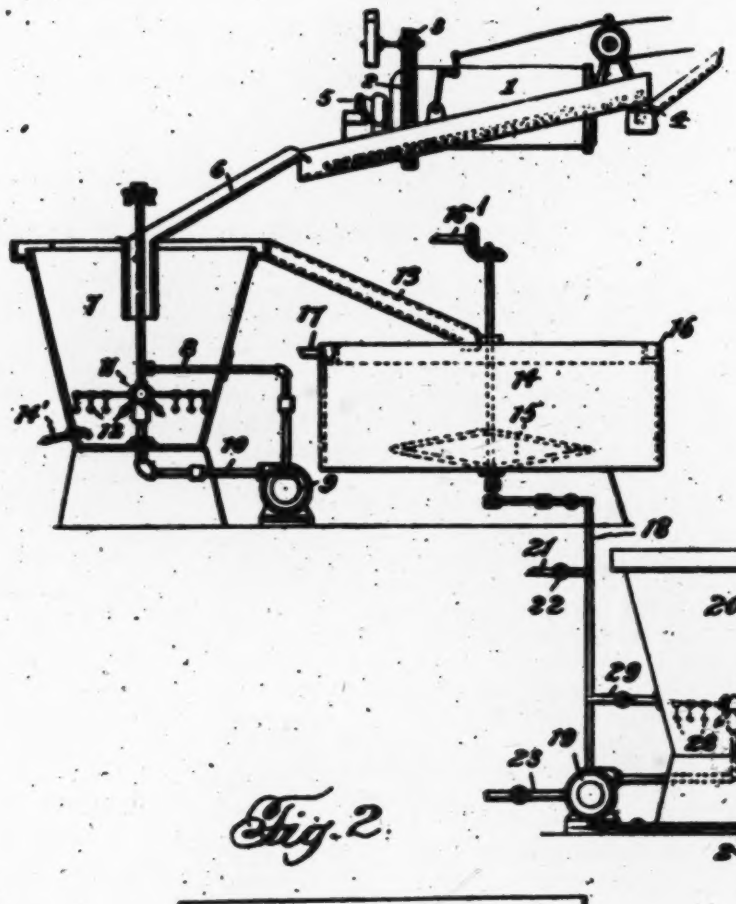


Fig. 1.

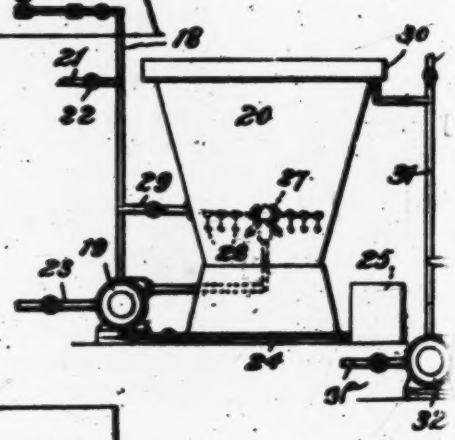
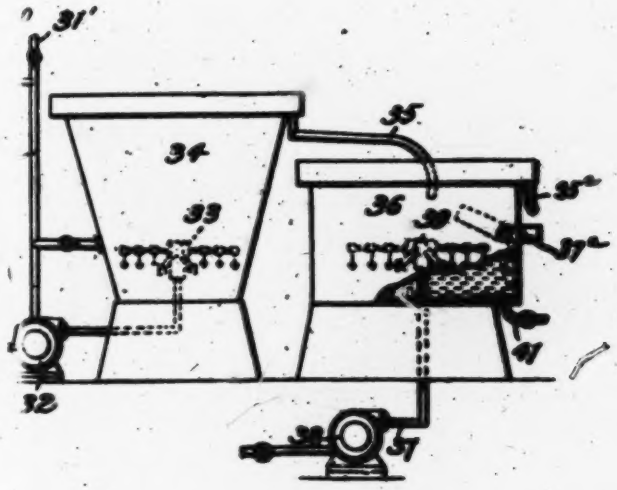
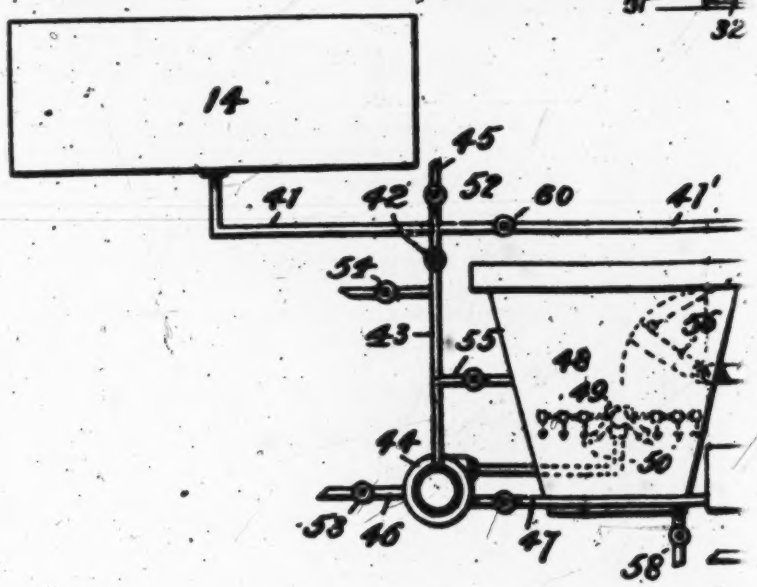


Fig. 2.







PATENT SPECIFICATION

151,236

Convention Date (United States): Sept. 16, 1919.

Application Date (in United Kingdom): Apr. 15, 1920. No. 10,573/20.

Complete Accepted: Aug. 15, 1921.

COMPLETE SPECIFICATION.

Improvements in Treating Carbonaceous Material.

I, WALTER EDWIN TREST, of 908, G. Street, City of Washington, District of Columbia, United States of America, Consulting Engineer, a citizen of the United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained and by the following statement:—

This invention relates to a process of separating carbonaceous materials suspended in a body of water.

More specifically, the invention has reference to the treatment of materials having a carbonaceous content, such as coal, lignite, *etc.*, in which the carbon or hydrocarbon content is mixed with non-combustible or ash-forming substances.

This invention is based on the discovery that where very finely pulverized coal suspended in water is treated by the subsequent addition of certain liquid hydrocarbons the latter will form with the carbonaceous content of the coal a dense agglomeration which will exclude the ash forming content, the water and slimes, and will precipitate itself in heavy globules or masses which may be easily removed from the liquid in which such ash and slimes remain, the ash being left exceptionally clean of carbonaceous matter. The liquid hydrocarbon preferably employed is liquid fuel oil, but tars, pitch and oil residuums which are solid at ordinary temperatures but sufficiently liquid when slightly heated may also be used to accomplish the reaction. The exact nature of the reaction is not entirely understood at present, but possibly may result from the non-miscibility of the water and liquid hydrocarbon, and the greater affinity existing between the liquid hydrocarbon and carbon particles than between said liquid and ash-forming particles or between the carbon particles and water. However this may be, the effect produced is a more complete and rapid separation than can be obtained by any other methods of which I am now aware.

In carrying out the present process, the coal is first reduced to a very fine state of division, preferably to an impalpable mass, in the presence of water to physically detach impurities from the carbonaceous matter. The reduced material suspended in the water is then treated with an agent such as liquid fuel oil or the like, which I have discovered will, upon proper agitation, agglomerate the carbonaceous material and expel the water and suspended impurities.

The quantity of oil used is based on the carbonaceous content of the pulverised material, the oil used being approximately 40%, by weight of the carbonaceous material. The process, therefore, not only aims to remove water from a saturated mass of carbonaceous material, but also contemplates the displacement or removal of impurities suspended in the water for which the treating agent has little or no affinity.

The agglomeration produced by this process is quite distinct in character from that produced by any oil flotation process, in that it is dense and heavy, free from air bubbles and slimes and is substantially free from water.

In the accompanying drawings,

Figure 1 is a diagrammatic, part-sectional side elevation of one form of apparatus for carrying out the process, this form, however, being given merely by way of example; and

[*Price 1/-*]

Figure 2 represents a slightly modified form.

In carrying out the process, the coal or other mineral containing both carbonaceous and non-carbonaceous material, after being coarsely ground and roughly separated from foreign impurities in the nature of slate, stones and the like, is then reduced to a very fine degree, varying somewhat according to the nature of the material undergoing treatment. The coal, however, is preferably reduced to an impalpable mass fine enough to pass through a 200-mesh or finer screen, to physically detach particles of ash from the particles of carbon. I preferably effect the reduction or grinding wet, which is by far the most expedient and inexpensive manner of finely reducing the materials, the coal being introduced to a tube mill 1 which is supported upon suitable trunnions and is rotated by means of the intermeshing gears 2 and 3 driven by any suitable source of power. The tube mill is provided with a feed trough 4 through which the material to be reduced is fed to the mill, and a discharge 5 leading to a hydraulic classifier for returning to the feed trough any material passing from the tube mill which has not been reduced to such a degree of fineness as to physically detach the inherent impurities of the coal from its carbonaceous content.

From the classifier, the water containing the suspended carbonaceous matter and detached impurities is discharged into a suitable trough 6 which supplies it to a separator 7 of any convenient construction. The separator is provided with a perforated pipe 8 at a point intermediate its ends leading through the wall thereof and connected with a pump 9 discharging into a pipe 10 in communication with a slowly-revolving agitator 11 having tangentially-disposed discharge nozzles 12 and operated by any suitable gearing. The pump serves to draw the water having suspended material therein through the pipe 8, and discharges the same into the separator through the nozzles 12, thus facilitating the separation of the heavier suspended impurities from the carbonaceous matter, particularly the iron sulphide, *etc.*, while the lighter carbonaceous material and any flocculent impurities which may happen to be present in suspension are carried away with the water, due to its current from the separator 7 by the discharge chute 13 and delivered to the collecting tank 14. The collected impurities in the separator 7 are removed therefrom, as they accumulate, through the discharge conduit 14^1.

In the tank 14 there is disposed a rake or stirrer 15 which is slowly revolved by means of the gearing 15^1, the carbonaceous material carried in suspension in the water settling in the tank when the water enters therein. This is due to the relatively large capacity of the tank 14 which causes the water, on entering, to lose its velocity, which will allow the carbonaceous material to settle, while flocculent impurities remain suspended in the water and overflow therewith into the launder 16 to be discharged through the pipe 17.

The material collected in the tank 14 has now been largely cleaned of its ash-forming content; but to effect a further removal of this ash, or to expel the water from the saturated, relatively-pure carbonaceous material, I then subject the material to the action of a liquid hydrocarbon, such as fuel oil, crude oil, navy oil, oil waste, oil residue, or other satisfactory agents. The mass collected in the tank 14 may be thinned by the addition of water and a proper agitation thereof; and I have found that if a mass of carbonaceous material suspended in water is treated with a suitable hydrocarbon agent, the oil will possess the characteristics of expelling the water and of agglomerating or collecting the carbonaceous matter. If any detached impurities, such as silica, silicates, or the like, exist in the carbonaceous mass, the oil not only agglomerates the carbonaceous material, but also expels the water and the said impurities, as the latter will associate with, or be carried in suspension in, the water and will not be agglomerated by the oil, the oil possessing a peculiar affinity for the carbonaceous matter and a repulsion for the water and its suspended impurities.

The collected mass in the tank 14 is discharged through the feed pipe 18, and is conveyed to a pump 19 which introduces it into a treatment chamber 20. The pipe 18 has communicating therewith an air-conducting pipe 21 controlled by a valve 22 to enable air to be supplied to the material, if such action is found to be desirable to facilitate a separation. The pump 19 also has a water-conducting pipe 23 leading thereto, and an oil conduit 24 communicating with a source of oil supply 25. The water with its suspended material is introduced directly into the treatment chamber 20, together with the treating oil, through the pipe 26 which communicates with a slowly

revolving agitator 27 having discharge nozzles 28; and the mass of material is then agitated therein so that the oil begins at this time to commingle with the carbonaceous material and to expel the water and suspended impurities therein. A conduit 29 leads from the treating chamber 20 back to the pump 19 to facilitate the agitation of the mass in said chamber.

It will be understood that the water containing the suspended impurities flows to the treatment chamber 20, but is not maintained therein for a sufficient period of time to bring about a complete agglomeration of the carbonaceous material. However, if desirable the material may be allowed to remain within the treating chamber 20 and sufficiently agitated to produce an agglomeration of the carbonaceous substances and exclusion of the water and suspended impurities. When the treating agent has become intimately commingled with the carbonaceous matter, the suspended material in the water flows into the launder 30 surrounding the top of the chamber, and then into a conveying pipe 31 leading to a pump 32 which is in communication with an agitator 33 disposed within a second treating chamber 34. After leaving the launder 30, the mass has become partially treated so that some of the water has given way to the oil, and a further agitation thereof in the treatment chamber 34 results in a greater liberation of the water and its suspended impurities and a further agglomeration of the carbonaceous material by the treating agent. Additional air may be introduced to the conduit 31 through the air conducting pipe 31', if this is found desirable, and likewise additional water may be supplied through the water feed pipe 31". The function of the two treating tanks 20 and 34 is to enable the treatment of the mass to be carried on continuously, it being manifest that the mass receives a partial treatment in the tank 20 which is carried out to a further extent in the tank 34. The agitator 33 is of the same construction as the agitator 27 in the first treatment chamber 20, and a detailed description thereof is not believed to be necessary.

From the treatment chamber 34, the partially agglomerated carbonaceous material and the water are delivered through the feed conduit 35 to a receptacle 36 in which the carbonaceous material and oil are thoroughly amalgamated, while the water and any impurities still carried in suspension therein are expelled from the amalgamated mass. The material, on entering the receptacle, will rapidly thicken or agglomerate therein because of the intimate commingling of the oil and carbonaceous matter; and when the agglomerated mass has been formed, additional water may be introduced into the receptacle through the water feed pipe 37 which communicates with a water pump 38 and which is provided with a water-distributing agitator 39 having a plurality of nozzles 40, the water being under sufficient pressure and in sufficient quantity to wash the mass without appreciably distributing the carbonaceous material. This additional water is not essential, but is used for effecting a further cleansing of the carbonaceous matter, the water washing away in suspension any impurities that may be incorporated therein. The agglomerated mass has little or no affinity for the water or for impurities, and rapidly gives up the impurities to the water to be carried away therein. The overflowing water carrying the impurities may be discharged through the discharge pipe 35', and also by the decanter 37', while the collected carbonaceous material may be removed through the discharge pipe 41, or lifted from the receptacle in any desired manner.

In affecting the agglomeration of the carbonaceous material and the expulsion therefrom of the impurities as well as of the water, various quantities of water may be employed, as this has little or no bearing upon the reaction. Likewise, the quantity of the oil or treating agent may vary depending upon the nature of the material undergoing treatment; but it is desirable not to employ too great a quantity of the oil or other treating agent, as the excess oil will not agglomerate or cooperate with the carbonaceous material, but will merely rise to the surface of the water in the treating receptacle and is subject to waste.

Too small a quantity of oil will not bring about the reaction; but where a limited amount of water is used and not enough oil to bring about the reaction, an agitation of the mass will cause the same to present a dry or solidified appearance and to become very soft and crumbly. The proper quantity of oil, however, may be easily ascertained in carrying out the process by adding the oil until the agglomeration of the carbonaceous material has taken place, and the suspended impurities and water segregate or are driven from the carbonaceous mass. The agglomerated mass is usually of a

heavy, greasy nature, the oil being thoroughly incorporated with the carbonaceous material. The agitation should be carried on until the reaction has occurred, and any suitable form of agitator may be used for supporting the process.

It will be understood that the impurities removed from the coal by my process are not only impurities of coal such as silica, silicates, iron sulphide, *etc.*, but also the inherent ligneous ash forming contents of the coal which so far as I am now aware, have not been separated from the carbonaceous content of coal by present-day methods. The carbonaceous material substantially freed of these troublesome inherent or natural impurities is susceptible of many uses, as it may be used as a fuel and will burn with but slight ash. The treating agent may also be removed therefrom, and the carbonaceous particles in a substantially dry state used as a powdered fuel.

In Figure 2 of the drawings I have disclosed a modification of the process wherein the suspended impurities in the water from the collecting chamber are introduced to suitable treating chambers and remain therein during the period of treatment, this being preferably an intermittent treatment as distinguished from a continuous treatment as disclosed in Figure 1. As shown in Figure 2, the material suspended in the water flows from the collecting tank or receptacle 14 through the feed pipe 41 controlled by the valve 42 into a pipe 43 leading to a pump 44. Air is likewise introduced to the pipe 43 by the air-feeding pipe 45, and water and oil are conveyed to the pump 44 through the water and oil feeding pipes 46 and 47 respectively, the oil being fed from a suitable oil tank A. The suspended mass is forced by the pump into the treating chamber 48 through the feed nozzles 50 of the agitator 49.

When the chamber or tank 48 has been supplied with a charge of the material and water, and suitable oil has been introduced thereinto, the valve in the oil-feeding pipe 47 is closed, and the valve 42 is likewise closed. The mass is then agitated being continuously drawn from the chamber 48 through the pipe 55 communicating with the pump 44 and then returned through the agitating nozzles 50 to the chamber 48. This operation is continued until a thorough intermingling of the oil or other treating agent and the carbonaceous matter has resulted, and an agglomeration of the carbonaceous matter and oil has taken place. When the material has become agglomerated, the water and suspended impurities will be excluded, and the latter may then be decanted by lowering a mechanically-operated decanting device 56 to a point where they will run out with the water through the discharge pipe 57. The agglomerated carbonaceous material and oil settle down in the tank or chamber, and may be withdrawn through the valve controlled outlet 58. Fresh water or other cleaning agent may be introduced into the tank for washing and carrying away any detached impurities therein.

The feed pipe 41[1] leading from the tank 14 extends to a second treating chamber 59, a suitable valve 60 being disposed in this pipe beyond the juncture of the pipe 41 therewith to enable the supply of material to either treating chamber to be opened or closed at will. The treating chamber 59 is of the same construction as the treating chamber 48, and as a detail description of that chamber has already been given, the same will suffice for the chamber 59.

I am aware that it has been proposed to treat graphitic ore by a method wherein the pulverized graphite ore is mixed with enough crude petroleum to thoroughly moisten it. The mixture is introduced into an apparatus which comprises a receptacle, with means for maintaining the constant flow of a body of water therethrough, a stirring and disintegrating apparatus, by means of which the aqueous suspension of the mass is effected, and a screen upon that side of the receptacle, preferably the bottom towards which the flow of water takes place, whereby the mingled carbon and oil is retained, while the gangue is carried with the water through the screen.

I have found however, that such is not applicable to coal and like carbonaceous material for separating the ligneous ash forming substances from the carbon since for the latter purpose it is essential that the finely divided coal or the like should be suspended in water previous to contact with the oil.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:

1. The process of treating materials having a carbonaceous content, more especially coal, lignite and the like which consists in subjecting the material in a fine state of subdivision suspended in water to the action of a hydrocarbon hav-

ing the property of forming a dense agglomerate with the carbonaceous particles to the exclusion of the water and non-carbonaceous particles.

2. A process according to Claim 1 in which the material is initially subjected to grinding or pulverizing action in the presence of water.

3. A process according to Claim 1 or 2 which the agglomerated mass after the original impurities and water have been separated therefrom, is further washed with a cleansing agent.

Dated this 15th day of April, 1920.

For the Applicant,
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Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.—1921.

UNITED STATES PATENT OFFICE.

HUGH RODMAN, OF EDGEWOOD, PENNSYLVANIA, ASSIGNOR TO RODMAN CHEMICAL COMPANY, A CORPORATION OF PENNSYLVANIA.

CASE-HARDENING MATERIAL.

1,076,235. Specification of Letters Patent. Patented Oct. 21, 1913.

No Drawing. Application filed May 1, 1913. Serial No. 764,880.

To all whom it may concern:

Be it known that I, HUGH RODMAN, a citizen of the United States, and a resident of Edgewood, in the county of Allegheny and State of Pennsylvania, have made a new and useful Invention in Case-Hardening Material, of which the following is a specification.

This invention relates to a new and useful material for carbonizing by dry-packing and to a new art of preparing it.

Many finely powdered materials which are chemically satisfactory are commercially unsatisfactory because of dustiness. This dustiness makes handling difficult, lowers the thermal conductivity and favors separation of particles of different size and density. On account of the difficulty in handling, as well as other objectionable features, it is desirable to bring the material together in the form of small masses of approximately uniform size and thereby eliminate the dust. Previous to this invention I have made use of dustlike material by transforming it into relatively large masses and then crushing the relatively large masses into smaller masses. In actual practice I have found that such treatment has some objection on account of the irregular section of the crushed small masses which contain edges and points. There is some difficulty in packing and settling uniformly in the pots and in use the edges and points easily break off and are reduced to dust.

One of the objects of my invention is to eliminate this dustiness and to increase the uniformity and thermal conductivity of the material. I have found that the result can be obtained by making the carbonizing material in the form of small pebbles or pills of approximately spherical shape, or at any rate, of such shapes that their edges will be rounded. This reduces the tendency to form dust, facilitates packing and serves to maintain a high thermal conductivity.

One method of producing the material in the desired form is as follows: Mix one hundred (100) parts of finely powdered coking coal (preferably fine enough to pass through a screen having at least 100 wires to the linear inch) with ten (10) parts of sodium carbonate and fifteen (15) parts of calcium carbonate or hydroxid. Moisten the dry mixture with a tacky binder, such, for example, as a solution of molasses in water, force the moist mass through screens having four wires to the linear inch, and then tumble in revolving barrels occasionally sprinkling in some dry mixture till rounded or compacted into firm pebbles. Undersize and oversize pebbles are then screened out and returned to the mixers while pebbles of approved size, which may be $\frac{1}{16}''$ or $\frac{1}{4}''$ in diameter, are dried with gentle heat, or, if desired, they may be coked by heating to about 1500° Fahrenheit. The molasses and carbonates or hydroxids react to form a bond which allows the coal to coke internally within the surface of each pebble, but prevents any deformation or disintegration of individual pebbles or adherence of adjacent pebbles, that is, each pebble cokes individually, binding and hardening all of the material within its own surface, but the pebble does not adhere, cohere or coke to adjacent pebbles.

Other methods of making approximately spherical small masses will occur to those skilled in the art.

Having thus described my invention, what I claim is:

1. As a new article of manufacture, case-hardening material consisting of small rounded masses of appreciable size.

2. As a new article of manufacture, case-hardening material consisting of small spherical masses of appreciable size.

3. As a new article of manufacture, case-hardening material in the form of small rounded pebbles, each pebble consisting of finely divided material held together by a tacky binder.

4. As a new article of manufacture, case-hardening material consisting of small rounded coked masses of appreciable size.

5. As a new article of manufacture, case-hardening material consisting of small rounded masses of appreciable size having a coking interior and a non-coking exterior.

6. As a new article of manufacture, case-hardening material consisting of small spherical masses of appreciable size having coking interiors and non-coking exteriors.

7. As a new article of manufacture, case-hardening material containing coking coal, consisting of small rounded masses.

8. As a new article of manufacture, case-hardening material containing coking coal, sodium carbonate and calcium carbonate, consisting of small rounded masses of appreciable size.

9. As a new article of manufacture, case-

hardening material in the form of small rounded pebbles, each pebble consisting of finely divided material held together by a tacky binder consisting of molasses.

10. As a new article of manufacture, case-hardening material in the form of small rounded pebbles, each pebble consisting of finely divided material containing coking coal held together by a tacky binder.

11. As a new article of manufacture, case-hardening material in the form of small rounded pebbles, each pebble consisting of finely divided material containing coking coal held together by a tacky binder consisting of molasses.

In testimony whereof, I have hereunto subscribed my name this 29th day of April, 1913.

HUGH RODMAN.

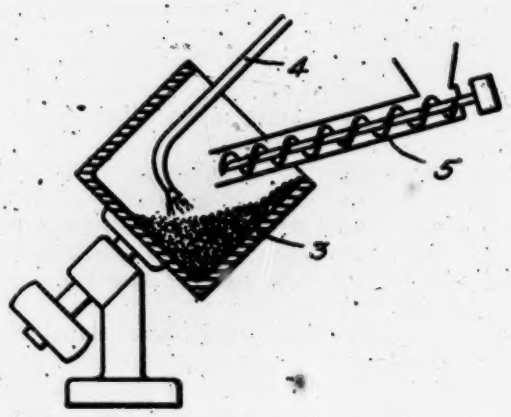
Witnesses:
C. W. McGhee,
Ada Romig.

H. RODMAN.

METHOD OF AGGLOMERATING FINELY DIVIDED MATERIAL.

APPLICATION FILED SEPT. 5, 1916.

1,239,221. Patented Sept. 4, 1917.



INVENTOR.

Hugh Rodman

BY Grew & McCauley

HIS ATTORNEYS IN FACT

UNITED STATES PATENT OFFICE.

HUGH RODMAN, OF EDGEWOOD, PENNSYLVANIA, ASSIGNOR TO THE RODMAN CHEMICAL CO., A CORPORATION OF PENNSYLVANIA.

METHOD OF AGGLOMERATING FINELY-DIVIDED MATERIAL.

1,239,231. Specification of Letters Patent. Patented Sept. 4, 1917.

Application filed September 5, 1916. Serial No. 118,472.

To all whom it may concern:

Be it known that I, HUGH RODMAN, a citizen of the United States, and a resident of Edgewood, in the county of Allegheny and State of Pennsylvania, have made a new and useful Invention in Methods of Agglomerating Finely-Divided Material, of which the following is a specification.

This invention relates to the pebbling or agglomerating of powdered material and discloses a new and improved process therefor.

In agglomerating or pebbling crushed or finely divided material by tumbling it with a binding agent, it has generally been necessary to employ batch methods and to screen out the oversize and undersize pebbles for reprocessing.

For instance pills have been made by alternately moistening and dusting small nuclei or centers till pills of the average size desired were produced. Over size and undersize pills were then removed by screening and the process repeated.

I secure a continuous process and avoid the screening of the batch process by using a tumbling or agitating device so operated that there is a marked grading action of the material being processed, and with the feed so arranged that the agglomerating or building up takes place only upon the undersize pills, the pills of full size meantime being out of the building up zone and, preferably, being continuously removed. In this way I secure a more efficient method of manufacturing and a far greater uniformity of product than can be gotten from the old batch method, or from a method in which the grading action is not taken advantage of.

The method I have described may be carried forward in several mechanical ways but the way I find best suited to the purpose is as follows:

A tub about two feet deep and two feet diameter with parallel sides is mounted to revolve upon its axis held at about thirty degrees from the horizontal. Finely divided material and binding agent are fed into this device by screw conveyer and sprinkler, or in any suitable way, care being taken to insure the binding agent falling only upon the material tumbling at the back or bottom of the tub. Nuclei may be added unless the tumbling material makes its own nuclei. The size and number of the nuclei formed by the tumbling mass may be regulated by varying the amount of binding agent added. The tumbling material rapidly and completely separates into a uniformly graded mass, all of the larger pills or agglomerates coming to the elevated or outer portion of the tub and grading from there, through smaller and smaller agglomerates, to the back or bottom of the tub where only the small agglomerates and finely divided material or dust remain. It follows that if the binding agent is applied only to the region or zone occupied by the very small agglomerates and dust there will be no building up of any but the small agglomerates and that these, as soon as they are larger than other agglomerates in the tub, will travel rapidly out of the building up zone. In this way the agglomerates are produced of very uniform size, only those being acted upon which are smaller than others.

As an improved method of manufacturing pills or agglomerates I make use of the process described to secure a continuous manufacturing method rather than a batch process, though the method of building up only the small size agglomerates may also be used as a batch method. For continuous manufacturing I use the device described, continuously feeding the binding agent into the back of the tumbling mass and continuously feeding the powdered material into the mass somewhat nearer the mouth of the tub and letting the finished pills or agglomerates continuously discharge over the lip of the tub. I find the best place to spray or sprinkle the binding agent is vertically over the lower edge of the bottom of the tub, and the best place to feed the powdered material to be about half way back from the mouth of the tub. Any excess powder will be quickly carried to the bottom of the tub and will not escape with the finished pills. Nuclei may be added if the material does not form its own.

I have used the device described to make small agglomerates or pills for carburizing steel, the powder being a mixture of finely divided carbonaceous materials and alkaline materials and the binding agent being molasses solution, glutrin, etc. I have also used the device to make agglomerates of other materials, as starch, macaroni flour, etc.

The grading action of the device described

is positive and rapid, but I am not able to give a satisfactory mechanical explanation for it. The action is much more positive than that secured by revolving the frustum of a cone upon its axis held in a horizontal position; and is different in that the cone frustum tends to carry the larger agglomerates to the bottom of the frustum, where the mass is deepest, while the inclined straight sided tub carries the larger agglomerates to the upper edge of the tub, where the mass is shallowest. I might add that the ordinary tumbling barrel made of the frustum of a cone and revolved upon its axis held at an angle of about thirty degrees from the horizontal, just about balances the two grading tendencies already described, so that there is little or no grading action obtained from these commercial machines. That is the shape of the device tends to force the larger agglomerates to the bottom of the frustum, while the angle of the axis tends to force them to the mouth and the two tendencies closely balance.

I believe that I am the first to make use of a process of making agglomerates or pills in which the agglomerates are graded as they are formed, and in which the smaller agglomerates only are subjected to the building up action. In other words I believe that I am the first to make agglomerates or pills, in which the agglomerates are graded as formed, the larger agglomerates being removed or moved out of the building up zone and the smaller agglomerates only being subjected to the building up action of the binding agent and dust or finely divided material.

I have shown in the accompanying drawing a device which may be employed in carrying out my improved process.

The device includes a tumbling barrel 3 having parallel sides and a diameter about equal to its depth. This is arranged to revolve upon its axis held at about 30° from the horizontal. A spraying or dripping device 4 is mounted so as to introduce a binding agent into the barrel and near the inner end thereof. Finely divided or powdered material, which may include nuclei if desired, is introduced into the barrel by means of apparatus such as a screw conveyer 5, which delivers the dry material at a point about midway between the inner end and the lip of the barrel. The larger agglomerates, as formed, move to the front of the barrel and, in a continuous process discharge over the lip of the barrel, whereas the dust and smaller agglomerates move to or remain at the inner end of the barrel and are eventually built up into agglomerates of such size that they move to the front of the barrel and are discharged. The final size of agglomerates discharged may be closely regulated by the number and size of nuclei added or formed. It will be apparent to those skilled in the art that other apparatus may be employed in carrying out my process.

What I claim is:

1. The method of building up pills or agglomerates which consists in agitating finely divided material and nuclei in a grading device and adding a binding agent only to the smaller of the agglomerates formed.

2. The continuous method of building up pills or agglomerates, which consists in agitating finely divided material and nuclei in a grading device, continuously adding a binding agent to the smaller agglomerates only, and continuously removing the larger agglomerates.

3. The method of building up pills or agglomerates which consists in tumbling finely divided material and nuclei in a tumbling and grading device and adding a binding agent only to the smaller of the agglomerates formed.

4. The continuous method of building up pills or agglomerates which consists in tumbling finely divided material and nuclei in a tumbling and grading device, continuously adding a binding agent to the smaller agglomerates only and continuously removing the larger agglomerates.

5. The method of building up pills or agglomerates which consists in tumbling finely divided material and nuclei in an inclined tumbling and grading device and adding a binding agent to the smaller agglomerates only.

6. The process of producing agglomerates, which consists in agitating dry finely divided material and nuclei, in adding a liquid binding agent to the agitated mass, causing the agglomerates to move away from the zone receiving the binding agent as they increase in size and finally discharging the agglomerates from the agitated mass.

7. The process of forming agglomerates, which consists in agitating finely divided material, continuously introducing a binding agent into a definite zone of agitation of the mass grading the agglomerates formed by causing them to move farther away from the zone receiving binding agent as they increase in size, and adding additional quantities of dry material to the agitated mass at a zone other than the zone receiving the binding agent.

8. The process of increasing the size of agglomerates which consists in agitating a mass of nuclei and finely divided material, introducing a binding agent to a definite zone of agitation of the mass and grading the agglomerates as they are formed, by moving them so that the smaller agglomerates only are subjected to the building up action of the dry material and binding agent.

9. The process of increasing the size of agglomerates, which consists in agitating a mass of nuclei and finely divided material, grading the agglomerates as they are formed by moving agglomerates of different sizes to different zones of agitation and subjecting the smaller agglomerates only to the building up action of a binding agent and finely divided dry material.

10. The method of producing pebbles which consists in agitating fine material and causing the larger agglomerates as formed to travel in one direction, while the smaller agglomerates and fine material travel in another direction, and subjecting the smaller agglomerates only to the action of a finely divided material and a binder.

11. The method of producing pebbles which consists in agitating fine material in a bed of varying depth and causing the larger pebbles as formed to travel toward the shallow edge of the bed, subjecting the deeper portion of the bed to the action of a binding agent, and removing the larger pebbles formed from the shallow edge of the bed.

12. The method of agglomerating material which consists in maintaining a mass of fine material in a bed of varying depth, adding a binding agent to the material at some point within the bed and thereby forming agglomerates, agitating the mass and causing a continuous flow of the smaller agglomerates and material toward the deeper portion of the bed and the larger agglomerates of material toward the shallower portions of the bed, in removing the larger agglomerates and wetting the smaller agglomerates only with binding agent while continuing the agitation.

In testimony whereof, I have hereunto subscribed my name this 2nd day of September, 1916.

HUGH RODMAN.

F. I. DU PONT.

PROCESS OF AND APPARATUS FOR POLISHING EXPLOSIVE POWDER.

APPLICATION FILED JULY 28, 1913.

1,239,368. Patented Sept. 4, 1917



Fig. 1.

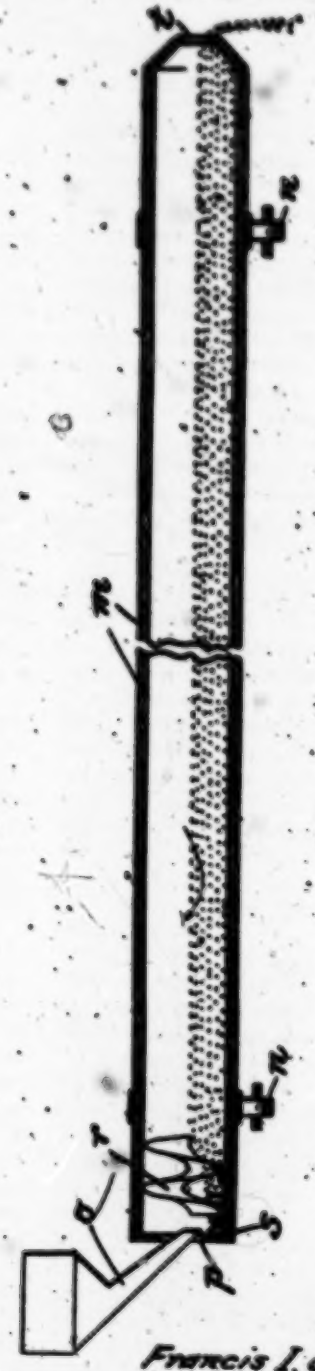


Fig. 2.

WITNESS:
Robt. Robertshaw.

INVENTOR
Francis I. du Pont
By Frank L. Bauer
ATTORNEY

UNITED STATES PATENT OFFICE.

FRANCIS I. DU PONT, OF WILMINGTON, DELAWARE, ASSIGNOR, BY DIRECT AND MESNE ASSIGNMENTS, TO BALL GRAIN EXPLOSIVES COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

PROCESS OF AND APPARATUS FOR POLISHING EXPLOSIVE POWDER.

1,239,368. Specification of Letters Patent. Patented Sept. 4, 1917.

Application filed July 28, 1915. Serial No. 42,966.

To all whom it may concern:

Be it known that I, FRANCIS I. DU PONT, a citizen of the United States, residing at Wilmington, county of New Castle, and State of Delaware, have invented a new and useful Improvement in Processes of and Apparatus for Polishing Explosive Powder, of which the following is a full, clear, and exact description, reference being had to the accompanying drawings, which form a part of this specification.

The object of my invention is to polish or glaze explosive granular powder. In the ordinary process of glazing powder, a large quantity—about one to two tons—is placed in a barrel from three to four feet in diameter and from six to eight feet in length. The barrel is filled from one-half to two-thirds full and is rotated on its axis at such a speed that the grains of powder slide over one another until they are sufficiently smoothened and polished. The operation is continued until all the powder is glazed.

The glazing operation is attended with considerable danger and not infrequently explosions occur. Such explosions invariably result in the total destruction of the mill and in the loss of the lives of any workmen who may be on the premises at the time of the explosion. No matter how carefully the operation is conducted, the danger of explosion remains.

Instead of attempting primarily to remove or diminish the danger of the ignition of the powder, to which efforts have hitherto been directed, my invention contemplates diminishing the destructive effects of the explosion to such an extent that, in the event of explosion, there will be practically no danger of injury either to the operatives or to the machinery. My invention also contemplates effecting this result while at the same time rendering the operation continuous and more nearly completely automatic and without reduction of output or sacrifice of efficiency but on the contrary with an actual increase in both these factors.

It is apparent that when a large amount of powder is in the form of a compact and confined mass, its ignition necessarily produces a violent explosion, whereas if the same quantity of powder were spread out over a great area or strung out in a long line, its ignition would cause it to burn rather than to explode with force, thereby immensely reducing its destructive effects. However, it is impracticable to thus distribute and mechanically handle, at any given time, a volume of powder that would be contained in a barrel six to eight feet long and three to four feet in diameter, while if a substantially smaller quantity were handled, the operation would not be feasible commercially.

In my invention I accomplish the results sought, without sacrifice of any essential factor, by so distributing and handling the powder that at any given time only a comparatively small quantity of powder is undergoing the polishing operation, while this smaller quantity of powder is so widely distributed that in the event of explosion no damage will result except practically negligible damage to the powder container. At the same time the speed of the operation is in inverse proportion to the quantity of powder at any given time undergoing treatment, thereby causing no decrease in output. Additionally, provision is made so that the operation may be conducted continuously instead of intermittently, thereby increasing the output, reducing labor, and diminishing the chances of accidental ignition.

The invention may be best explained by first referring to the accompanying drawings, in which—

Figure 1 is a cross-sectional diagrammatic view of my improved apparatus for carrying out my improved process. Fig. 2 is a longitudinal sectional view of the same.

In place of the usual barrel I provide a tube *m* which, for example, may be eight inches in diameter and thirty-two feet long. The tube is revolved as on rolls *n*. A feed chute *o* delivers into an opening *p* in one end of the tube and at this end there is a short section of a spiral *r* fixed in the tube and flaring in the direction to start the material moving toward the other end of the tube. A free space at *s* is left between the spiral and the flange at the end of the tube, into which space the material is fed, and even though the level at this point is low, the effect of the spiral is to fill the tube continuously to as much higher level as may be desired. This level is regulated by the diameter of the hole *t* at the delivery end of the tube. Thus by continuously supplying explosive

powder through the feed chute *o* and revolving the tube *m* at a certain speed, the material is gradually fed along throughout the length of the tube and overflows out of the opening *t*, maintaining an active polishing or glazing zone the full length of the tube.

I have discovered that, in any polishing barrel, the powder actually being acted upon at any given time is not the entire mass of powder in the barrel but only a comparatively thin layer. Thus, Fig. 1 represents the action of the material in a polishing barrel, which is rotating in the direction of the arrow. The material climbs up the rising side of the barrel and when the particles have reached a point well up toward the top, they tumble down the exposed face. It is only in this tumbling down operation that the grains rub or slide upon one another and receive a polish, and it is only in the comparatively thin zone represented at *x—y* that this operation occurs. Thus the amount of powder being glazed at any given time depends upon the area of the active zone (assuming a given speed of movement of the sliding grains) and not upon the bulk of powder in the container.

For example, it may readily be calculated that a barrel 6 feet long and 4 feet in diameter will contain six times as much powder as a tube 8 inches in diameter and 36 feet long, but the area of the active zone, assuming that both containers are half full, will be as great in the tube of small capacity as in the barrel of large capacity. If, therefore, the tube is rotated at such greater angular speed than the barrel as to impart to the individual grains the same desirable speed of travel as in the barrel, it is clear that, at a given time, as much powder is being glazed in the tube as in the barrel and that the entire contents of the tube will be glazed in one-sixth the time required to glaze the entire contents of the barrel.

It will thus be seen that within any given time the output of my improved polishing apparatus will be as great as the output of the ordinary glazing barrel, assuming no loss of time in filling and discharging the barrel. In fact, however, this loss of time is not a negligible factor and therefore the output of my improved apparatus may actually be increased, if desired, over that of the ordinary polishing barrel, while at any given time a very much smaller quantity of powder is being glazed and the same is so widely distributed that, in the event of ignition, it will burn like a fuse rather than explode like a confined charge. Indeed, actual experience shows that the only effect is to spread apart the staves of the tube to allow the flame to escape, and, aside from the charring of the wood work by the burning powder, the machinery of the mill is not injured at all.

While, therefore, it is essential to my invention that the length of the powder mass be great relatively to its depth, and while it is clear that if the tube is half filled, such depth will approximate the radius of the tube, it will be understood that if the tube be made of substantially greater diameter and contain only the same quantity of powder the results will be the same. The essential factor is, therefore, the relation between length and depth of powder mass. Experiment has shown that, in the event of ignition of the powder, burning with flame, as distinguished from explosion with destructive force, cannot be assured, in a commercial plant, unless the ratio between length and depth of powder mass is at least 14 to 1, with a maximum depth of eight inches of powder mass; or, in the event that the tube is about half filled, its diameter must not exceed 16 inches, while the ratio between its length and radius should be at least 14 to 1.

It will be understood that the substitution of the elongated tube of relatively small capacity having a comparatively rapid angular speed of rotation for the compact barrel of relatively large capacity having a relatively slow angular rotation is not a mere change in degree, because the results are different in kind, in that in the one case the operation is attended with the greatest danger of destruction of life and the entire mill whereas in the other case this danger is not merely reduced but is entirely eliminated and the operation rendered absolutely safe.

It will be understood that the invention is applicable either to the smoothening of powder preparatory to final glazing with graphite or other polishing material, or to the final glazing after the smoothening operation.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:

1. The process of polishing explosive granular powder which consists in distributing the powder longitudinally to form an elongated string, imparting to the powder a movement of rotation on an axis parallel to its forward progressing movement, applying a forward propulsion force directly to the powder throughout only a relatively small part of its length and restricting the advance of the powder at the far end of the string to regulate and maintain its depth.

2. The process of polishing explosive granular powder which consists in distributing the powder longitudinally to form an elongated string, continuously supplying powder at the beginning of the string, applying a forward propulsion force to the powder at approximately the beginning of the string and throughout a relatively short

length of the string, imparting to the powder a movement of rotation on an axis parallel to its forward progressive movement while eliminating all obstruction to the free rolling of the grains one upon another, and regulating the depth of the string independently of the supply and speed of rotation.

3. The process of polishing explosive powder which consists in distributing the powder in a substantially horizontal elongated string, applying a forward propulsion force to the powder at one end of the string, imparting to the powder a movement of rotation on an axis parallel to its forward progressive movement while eliminating all obstruction to the free rolling of the grains one upon another, and restricting the advance of the powder at the other end of the string to regulate its depth.

4. The process of polishing explosive granular powder which consists in distributing the powder longitudinally to form a string having a length not less than approximately fourteen times its maximum depth, imparting to the powder a turning movement on an axis parallel to its longitudinal direction of extension, and controlling the desired depth by restricting its longitudinal flow.

5. In an apparatus for polishing explosive powder, the combination with a tube of comparatively great length and small diameter, its interior, throughout the greater part of its length, being open and free of obstruction to tumbling of the powder, of means to feed the powder into one end of the tube, and means occupying a relatively short part of the length of the tube to insure the forward travel of the powder within the tube.

6. In an apparatus for polishing explosive powder, the combination with a tube of comparatively great length and small diameter, of means to rotate the tube, feeding means to introduce the powder at one end thereof, and means within the tube and occupying a relatively short part of its length and positioned at the inlet end thereof to start the powder through the tube.

7. In an apparatus for polishing explosive powder, the combination with an elongated rotary tube, of feeding means to introduce the powder at one end, propulsion means occupying only a relatively short part of the length of the tube to effect the propulsion of the powder toward the discharge end, and means controlling the discharge and determining the depth of the material in the tube.

8. In an apparatus for polishing explosive powder, the combination with a tube of comparatively great length and small diameter, of means to rotate the tube, feeding means to introduce the powder at one end thereof, propulsion means occupying a relatively short part of the length of the tube and positioned at the inlet end thereof, and means at the discharge end of the tube to regulate the depth of powder therein.

9. In an apparatus for polishing explosive powder, the combination with an elongated rotary tube, of means to introduce the powder at one end thereof, a section of a spiral at the inlet end portion of the tube and turning therewith, the discharge end of the tube having a central hole whose diameter regulates the depth of powder in the tube.

10. In an apparatus for polishing explosive powder, the combination with an elongated rotary tube, having a central opening at its inlet end, of a hopper feeding into the inlet end thereof, and a section of a spiral fixed to the tube at its inlet end portion but spaced from the extreme inlet end, the discharge end of the tube being conical and provided with a central opening.

11. In an apparatus for polishing explosive powder, the combination with an elongated rotary tube, means to introduce the powder at one end thereof, means shorter than the tube positioned therein adjacent the inlet end thereof and adapted to impart a forward movement to the powder, a discharge opening at the other end of the tube, the diameter of which relative to the speed of forward movement of the powder regulates the depth of the powder in the tube.

In testimony of which invention, I have hereunto set my hand, at Philadelphia, on this 27th day of July, 1915.

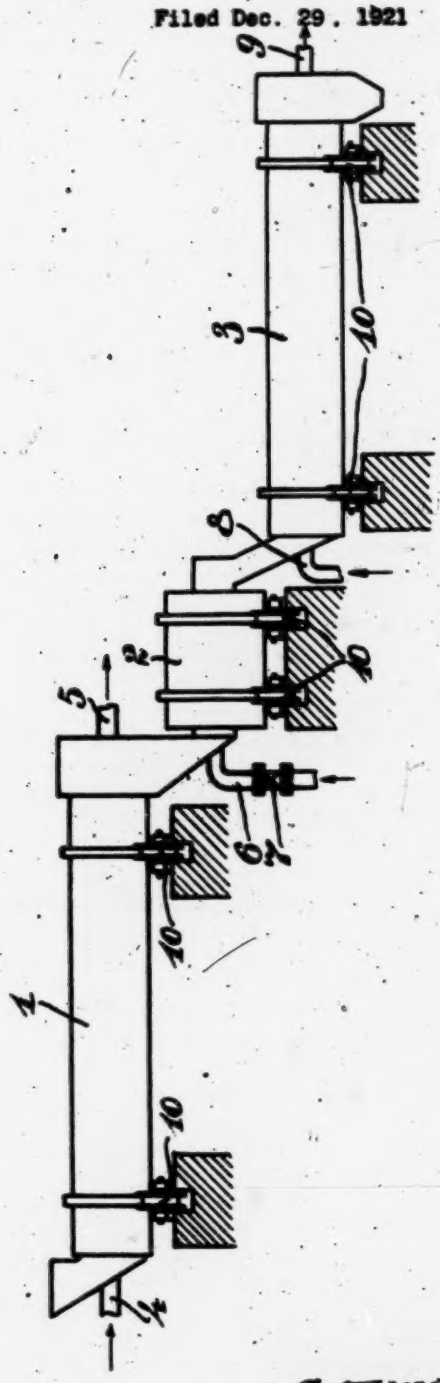
FRANCIS I. DU PONT.

Aug. 28, 1923. 1,466,571

C. T. THORSSELL ET AL

PROCESS FOR AGGLOMERATING PULVEROUS MATERIALS

Filed Dec. 29, 1921



Inventors
C. T. Thorssell
O. Troell
by

Patented Aug. 28, 1923. 1,466,571

UNITED STATES PATENT OFFICE.

CARL THEODOR THORSELL AND OTTO TROELL, OF GOTTENBORG, SWEDEN, ASSIGNORS TO AKTIEBOLAGET KVAFVEINDUSTRI, OF GOTTENBORG, SWEDEN.

PROCESS FOR AGGLOMERATING PULVEROUS MATERIALS.

Application filed December 29, 1921. Serial No. 525,709.

To all whom it may concern:

Be it known that we, CARL THEODOR THORSELL and OTTO TROELL, citizens of Sweden, residing at Gottenborg, Sweden, have invented certain new and useful Improvements in Processes for Agglomerating Pulverous Materials (for which I have made application for patents in Sweden, filed January 26, 1921, and in Great Britain, filed February 2, 1921), of which the following is a specification.

This invention relates to a process for agglomerating pulverous material, especially such material as is used for the absorption of nitrogen. When nitrogen is to be absorbed in mixtures of carbon and alkali metals, or alkaline earth metals, or their compounds, several disadvantages are associated with the use of pulverous mixtures.

In such processes for instance, pulverous mixture, has a tendency to bake together on account of the high temperature of reaction in relation to the melting point of the alkali compound used. It is obvious that this renders difficult the removal of the mass from the vessel of reaction, and also results in uneven distribution of the nitrogen thereby reducing the output of the reaction. Also if the mass in the vessel of reaction is subjected to a sudden increase of pressure in such manner that the pulverous material is ejected the powder will immediately catch fire and be the cause of fatal accidents. Granular material has therefore been used instead of powder, and experiments have shown that the use of hard grains or pieces of spherical or approximately spherical form is to be preferred, as such balls possess a greater resistance against being rubbed to pieces and do not form any dust. On the contrary, they will leave the vessel of reaction, for instance a continuously working shaft furnace, without altering their form. In spite of their hardness such grains or balls allow the penetration of quite a sufficient quantity of nitrogen, as they present large surfaces to the gas, whilst their own contact surfaces are small.

When such pulverous mixtures are agglomerated or granulated the mixture is first moistened and then worked up mechanically in a moist state to form grains or sticks. It has been proposed to manufacture such granular or stick-shaped material by pressing the mixture in apparatuses similar to brick making machines or sausage-grinders, but this method has proven to be too costly. The productiveness of such apparatuses is comparatively trifling in relation to the cost and the consumption of power is very considerable, as the material must be subjected to a very vigorous pressing through rather fine holes. The form of the agglomerated material is also unsuitable as it is very difficult to produce any forms other than sticks. The most suitable form, however, is hard balls, as indicated above.

The present invention relates to a process for agglomerating materials, whereby a considerable output is obtained with a small consumption of power. Further, the finished agglomerated material is dried at the same time as the material to be agglomerated is exposed to a preliminary drying.

When nitrogen is to be absorbed the mixture of carbon and compounds of alkali metals, or alkaline earth metals, with or without catalyzers is heated in furnaces, whereby cyanide is formed. After that the material is transferred into autoclaves in which it is treated with steam under pressure for the purpose of forming ammonia and regenerating carbonate (or the forming of formiate).

A suitable quantity of carbon is added to the carbonate or formiate removed from the autoclaves, whereafter the mixture is agglomerated and again carried back to the furnace of reaction. The percentage of water in the mixture to be agglomerated cannot be kept perfectly fixed and is usually greater than is suitable for the agglomerating operation. Even if this is not the case it is best to keep the percentage of water somewhat greater than is necessary and afterwards to adjust the same in a drying apparatus to the percentage most suitable for the material in question. The fact is that a different degree of wetness is required at the agglomerating operation for different materials.

According to the invention therefore the material containing more water than is suitable for the agglomerating process is first dried in such a way that it attains a proper degree of wetness. This is preferably done in one or more rotating cylinders or the like which are heated internally by means of burning gases passing through the same, preferably in the same direction as the material in order to lessen the burning up of

the coal in the material as much as possible. In this drying apparatus balls commence to be formed which however are rather irregular in shape and soft.

From this drying apparatus the mixture is carried to another arrangement of rotating cylinders for the agglomerating proper. The degree of wetness must here be kept constant and this is done by passing gases containing steam through these cylinders. In addition steam may be admitted directly to this apparatus for regulating the degree of wetness by connecting the cylinders with a steam pipe through adjustable valves or the like. In this apparatus the balls prepared in the drying apparatus mentioned above develops into hard, almost perfectly spherical grains.

After this the grains or balls thus finished are carried to a final drying apparatus which also preferably consists of one or more rotating cylinders internally heated by burning gases which are preferably passed through the cylinders in the same direction as the material. The drying operation easily takes place on account of the spherical form of the material, whereby a large surface is presented to the hot gases. These gases having passed through the drying apparatus and there taken up a quantity of steam are then wholly or in part carried to the intermediate apparatus where the agglomerating proper takes place and help there to keep the percentage of water constant. The dust eventually carried away by these gases from the drying apparatus sticks fast to the balls in the agglomerating system and is worked into them.

A simple form of apparatus for carrying out the process herein disclosed is illustrated in the accompanying drawings, in which, the figure represents diagrammatically the apparatus employed.

In the illustration 1 represents the first rotating cylinder, in which the material is dried to a certain degree; 2 represents the second cylinder in which the degree of wetness is kept approximately constant and 3 is the third cylinder in which the material is finally dried. A pipe 4 is provided to supply burning gases to cylinder 1, which burning gases escape through outlet 5 from said cylinder 1. Steam is supplied to cylinder 2 by means of a pipe 6, a valve for controlling the steam being indicated at 7. Burning gases are supplied to the drying cylinder 3 by a pipe 8 and permitted to escape from said cylinder through pipe 9. Cylinders 1, 2, and 3 are supported for rotation, such being here indicated by rollers 10 on which the cylinders are supported, with the rollers arranged to be operated in any usual or preferred manner.

What we claim is:

1. Process for agglomerating pulverous materials consisting in passing the material through two systems of heated, rotating cylinders, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system is carried to a third system consisting of one or more heated, rotating cylinders and there dried.

2. Process for agglomerating pulverous materials consisting in passing the material through two systems of rotating cylinders internally heated by burning gases, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system is carried to a third system consisting of one or more rotating cylinders internally heated by burning gases and there dried.

3. Process for agglomerating pulverous materials consisting in passing the material through two systems of heated, rotating cylinders, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system is carried to a third system consisting of one or more heated, rotating cylinders and there dried, steam being introduced in adjustable quantities into the second system for the purpose of regulating the degree of wetness.

4. Process for agglomerating pulverous materials consisting in passing the material through two systems of rotating cylinders internally heated by burning gases, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system is carried to a third system consisting of one or more rotating cylinders internally heated by burning gases and there dried, the gases leaving the third system being passed wholly or partly to the second system in order to keep the degree of wetness constant.

5. Process for agglomerating pulverous materials consisting in passing the material through two systems of rotating cylinders internally heated by burning gases, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system

is carried to a third system consisting of one or more rotating cylinders internally heated by burning gases and there dried, the heating gases being passed through the first system in the same direction as the material to be treated.

6. Process for agglomerating pulverous materials consisting in passing the material through two systems of rotating cylinders, internally heated by burning gases, in the first of which systems the material is dried to a certain degree of wetness whilst the degree of wetness is kept approximately constant in the second system, where the agglomerating proper takes place, whereafter the material agglomerated in the second system is carried to a third system consisting of one or more rotating cylinders internally heated by burning gases and there dried, the heating gases being passed through the third system in the same direction as the agglomerated material.

In testimony whereof, we have signed our names to this specification in the presence of two subscribing witnesses.

CARL THEODOR THORSSELL.
OTTO TROELL.

Witnesses as to signature of Carl Theodor Thorssell:

E. JOHNSON,
A. T. LUNCY.

Witnesses as to signature of Otto Troell:

G. PETERSSON,
PHILIP WILKONS.

[fols. 471-480] PLAINTIFF'S EXHIBIT No. 8

UNITED STATES DEPARTMENT OF THE INTERIOR
Harold L. Ickes, Secretary
BUREAU OF MINES
R. R. Sayers, Acting Director

CARBON BLACK

BY G. R. HOPKINS AND H. BACKUS

Chapter (preprint) from
MINERALS YEARBOOK 1940
REVIEW OF 1939

UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON : 1940

After this publication has served your purpose and if you have no further need for it, please return it to the Bureau of Mines, using the official mailing label on the last page

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The complete volume, covering all mineral commodities, may be
purchased from the Superintendent of Documents,
Washington, D. C., after August 1940

CARBON BLACK

By G. R. Hopkins and H. Rackus

SUMMARY OUTLINE

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Under the stimulus of the war and the recovery in rubber production the carbon-black industry reached record levels in all phases of

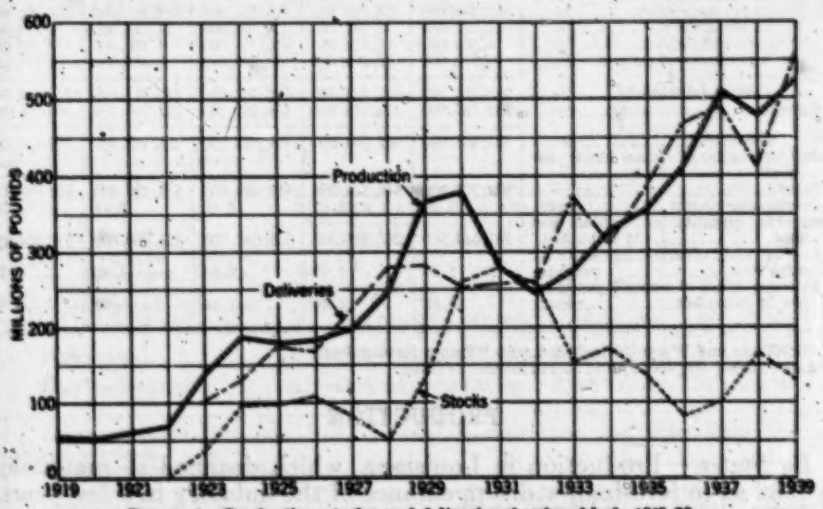


FIGURE 1.—Production, stocks, and deliveries of carbon black, 1919-39.

production and consumption in 1939. The output was 525,166,000 pounds, 10 percent more than in 1938, and sales totaled 560,533,000 pounds, 36 percent more than in 1938. The position of stocks improved considerably, but prices continued distressingly low. The domestic price rallied slightly, but the trend of export quotations continued downward. The average f. o. b. price rose from 2.41 cents per pound in 1938 to 2.45 cents in 1939—about enough to cover the increase in the average price of gas from 0.89 cent per thousand cubic feet in 1938 to 0.94 cent in 1939. Increases up to a quarter of a cent on domestic quotations on April 1, 1940, were heartening.

Figure 1 shows the sharp recovery in production and deliveries from the slump of 1938.

Salient statistics of carbon black produced from natural gas in the United States, 1935–39

	1935	1936	1937	1938	1939
Number of producers reporting	21	20	24	21	22
Number of plants	54	54	57	55	49
Quantity produced:					
By States and districts:					
Louisiana.....pounds	64,875,000	89,201,000	66,361,000	39,534,000	51,734,000
Texas:					
Panhandle district.....do	263,361,000	221,576,000	305,247,000	382,368,000	410,130,000
Rest of State.....do	[1] 24,513,000	12,330,000	15,821,000	34,735,000	43,044,000
Total Texas.....do	[1] 287,874,000	233,906,000	321,068,000	417,104,000	453,174,000
Other States.....do	(2)	18,236,000	23,157,000	20,401,000	20,256,000
Total United States.....do	352,749,000	411,343,000	510,606,000	477,039,000	525,164,000
By processes:					
Channel process.....do	316,284,000	366,876,000	444,427,000	441,304,000	464,388,000
Other processes [3].....do	36,465,000	44,468,000	66,179,000	35,735,000	60,776,000
Stocks held by producers Dec. 31 pounds	136,086,000	79,562,000	100,497,000	186,159,000	130,792,000
Losses.....do	936,000	113,000	76,000	[4] 65,000	
Quantity sold:					
Domestic deliveries:					
To rubber companies.....do	213,708,000	278,018,000	299,894,000	217,281,000	316,621,000
To ink companies.....do	15,177,000	17,787,000	18,116,000	14,131,000	21,929,000
To paint companies.....do	6,550,000	6,914,000	6,189,000	4,229,000	6,382,000
For miscellaneous purposes.do	9,916,000	10,299,000	11,803,000	7,983,000	11,773,000
Total domestic sold.....do	245,351,000	313,018,000	336,002,000	243,474,000	356,705,000
Export.....do	142,185,000	154,718,000	184,253,000	167,968,000	203,828,000
Total sold.....do	387,536,000	467,736,000	489,615,000	411,442,000	560,533,000
Value (at plants) of carbon black produced:					
Total	$13,755,000	$16,110,000	$17,389,000	$11,496,000	$12,857,000
Average per pound.....cents	2.90	2.92	2.41	2.41	2.45
Estimated quantity of natural gas used.....M cubic feet	241,589,000	283,421,000	341,085,000	234,950,000	347,270,000
Average yield of carbon black per M cubic feet.....pounds	1.46	1.45	1.50	1.47	1.51
Average value of natural gas used per M cubic feet.....cents	1.37	1.30	1.26	.89	.94

[1] Oklahoma and Wyoming included with "Texas: Rest of State."
[2] Lewis, roller, "Special," and thermatomic. [3] Gain.

PRODUCTION

By States.—Production in Louisiana, which declined so materially in 1938 as to foreshadow disappearance of the industry in a few years, scored a notable come-back in 1939, rising to 51,734,000 pounds compared with 39,534,000 in 1938. Production in Kansas and Oklahoma continued at about 20 million pounds. Texas production rose to a new peak of 453,174,000 pounds—9 percent (36,070,000 pounds) above 1938. In 1939 the output in the Panhandle was 410,130,000 pounds, equivalent to 91 percent of the State total compared with 92 percent in 1938. Wyoming, which has been a producer since the first statistics were collected in 1919, ceased to produce in 1939.

By months.—According to estimates based upon monthly figures of the National Gas Products Association, the monthly output of carbon black averaged just above 40 million pounds until the last quarter, when under the stimulus of the war it increased to nearly 50 million pounds.

Carbon black produced from natural gas in the United States, 1935–39, by States

Year	Production (thousands of pounds)				Average value per pound (cents)
	Louisiana	Texas	Other States	Total	
1935	64,875	[1] 267,874	(1)	332,749	3.90
1936	59,201	333,906	[2] 18,238	411,345	3.92
1937	66,391	421,068	[3] 23,157	510,606	3.41
1938	39,534	417,104	[3] 20,401	477,039	2.41
1939	51,734	455,174	[4] 20,258	525,166	2.45

[1] Oklahoma and Wyoming included with Texas.
[2] Oklahoma and Wyoming.
[3] Kansas, Oklahoma, and Wyoming.
[4] Kansas and Oklahoma.

Carbon black produced from natural gas in the United States in 1939, by States and by major producing districts

State and district	Producers reporting [1]	Number of plants	Production			Natural gas used			
			Pounds	Value at plant		M cubic feet	Average yield per M cubic feet (pounds)	Value	
				Total	Average cents			Total	Average per M cubic feet (cents)
Kansas	1	1	(2)	(2)	(2)	(2)	(2)	(2)	(2)
Louisiana: Monroe-Richland district (Morehouse and Ouachita Parishes)	6	7	51,734,000	$1,271,000	2.62	21,777,000	2.38	$631,000	2.90
Oklahoma	3	3	[2] 20,258,000	[2] 529,000	[2] 2.61	[2] 11,572,000	[2] 1.71	[2] 163,000	[2] 1.37
Texas:									
Panhandle district (Carson, Gray, Hutchinson, Moore, and Wheeler Counties)	18	31	410,130,000	9,492,000	2.31	287,880,000	1.42	2,324,000	.81
Rest of State (Nueces, Stephens, Ward, and Winkler Counties)	5	7	43,044,000	965,000	2.24	25,741,000	1.67	145,000	.56
Total, Texas	[1] 18	38	453,174,000	10,457,000	2.31	313,621,000	1.44	2,469,000	.79
Total United States	[1] 22	49	525,166,000	12,857,000	2.45	347,270,000	[1] 1.51	3,263,000	.94

[1] In counting the total number of producers a producer operating in more than 1 State, district, or county is counted only once.
[2] Kansas included with Oklahoma.

Carbon black produced from natural gas in the United States in 1939, by months, in pounds

Month	National Gas Products Association	Bureau of Mines [1]		Month	National Gas Products Association	Bureau of Mines [1]	
		Total	Daily average			Total	Daily average
January	37,313,345	41,393,147	1,341,714	August	35,948,556	40,070,166	1,292,586
February	34,124,815	36,074,535	1,388,805	September	40,038,362	44,639,110	1,487,970
March	39,720,197	44,234,010	1,426,897	October	42,450,401	47,317,457	1,526,370
April	38,239,668	42,745,812	1,424,950	November	42,204,448	47,159,907	1,571,997
May	40,217,988	44,954,210	1,450,136	December	43,254,022	48,362,755	1,559,963
June	37,296,775	41,606,181	1,386,939				
July	39,748,355	44,236,010	1,426,807		470,495,909	525,166,000	1,438,811

[1] Monthly figures obtained by allocating the Bureau's annual total proportionately to the association's monthly data.

Methods and yields.—Production by processes other than channel increased materially in 1939, nearly compensating for the loss in 1938. The output by "other" processes in 1939 amounted to 60,578,000 pounds, equivalent to 12 percent of the total, compared with 35,755,000 pounds comprising only 7 percent of the total in 1938.

The yield of carbon black rose from 1.47 pounds per thousand cubic feet in 1938 to a new peak of 1.51 pounds in 1939. This gain was due to the rise in relative importance of "other" processes, several of which give unusually high yields.

Despite the low price level, research in the fundamentals of carbon-black production and consumption continued, as it is quite generally recognized that the behavior of different blacks under varying conditions is a fertile field for experiment. One of the products of this research ("dustless" carbon black) increased steadily in popularity in 1939; it is doubtful if more than 10 percent of the present output is of the old nongranular type.

Number and capacity of plants.—New construction continued at a low ebb in 1939. About a half dozen plants were dismantled, and only two new ones were added to the operating list.

The total daily capacity of the operating plants, which remained virtually at a standstill in 1938, declined from about 1,736,000 pounds in 1938 to about 1,714,000 in 1939. The capacity in Texas increased about 30,000 pounds, but this was outweighed by a decline of about 70,000 pounds in Louisiana. The operating ratio, or the ratio of daily average production to average capacity, increased from 75 percent in 1938 to 84 percent in 1939.

Number and daily capacity of carbon-black plants operated in the United States, 1938–39, by counties or parishes.

State	County or parish	Number of plants		Total daily capacity (pounds)	
		1938	1939	1938	1939
Kansas	Grant	1	1	(1)	(1)
Louisiana	Morehouse	2	1	33,850	12,000
	Ouachita	9	6	225,775	168,995
		11	7	249,625	180,995
Oklahoma	Beckham	1	1		
	Seminole	1	1	[1] 76,750	[1] 91,750
	Texas		1		
		2	3	[1] 76,750	[1] 91,750
Texas	Carson	[2] 1	[2] 1		
	Moore	6	6	413,700	411,500
	Wheeler	2	2		
	Gray	10	8	340,360	311,180
	Hutchinson	[2] 14	[2] 14	[2] 548,120	[2] 567,670
	Eastland	1			
	Nueces		1		
	Stephens	4	4	107,300	150,000
	Ward	1	1		
	Winkler	1	1		
Wyoming	Niobrara	40	38	1,409,480	1,441,120
		1		(1)	
United States		55	49	1,735,855	1,713,865

[1] 1938: Kansas and Wyoming included with Oklahoma; 1939: Kansas included with Oklahoma.
[2] 1 plant, in both Carson and Hutchinson Counties, counted in Hutchinson County.

Producers.—The latest complete list of carbon-black producers (as of December 31, 1937) was given on page 967 of Minerals Yearbook, 1938. Changes in the list for 1938 were given on page 1073 of Minerals Yearbook, 1939. The changes in 1939 are as follows: The Southern Carbon Co. discontinued operations, and its three Louisiana plants at Fowler, Perryville, and Swartz were dismantled; the J. M. Huber Corporation closed its plants in Louisiana and Wyoming permanently; the Columbian Carbon Co. closed its plant at Pampa but opened one at Corpus Christi; and the General Atlas Carbon Co. began operations at its plant near Guymon, Okla.

DEMAND

Total deliveries.—Sales of carbon black in 1939 totaled 560,533,000 pounds—36 percent more than in 1938. Domestic sales in 1939 were

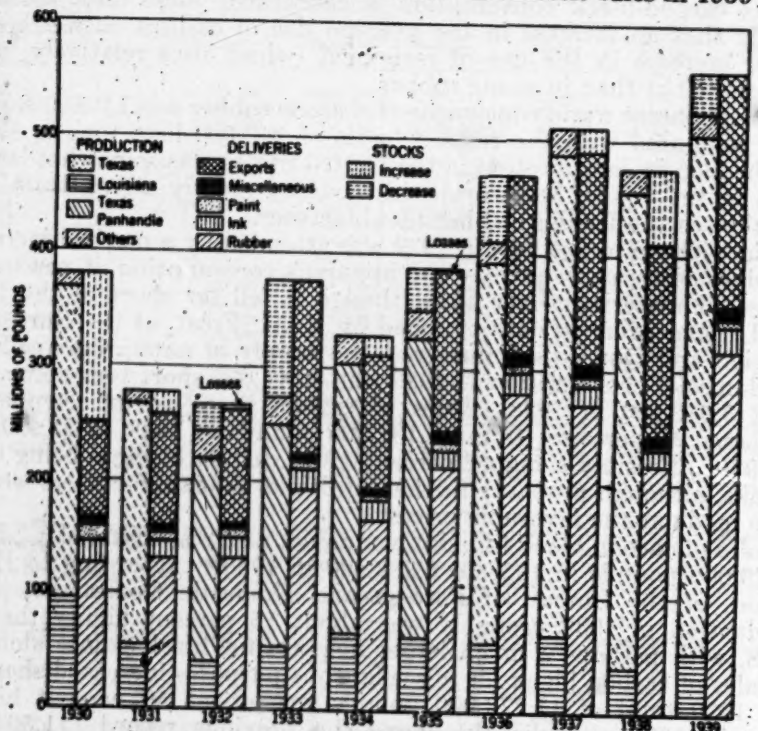


FIGURE 2.—Production and consumption of carbon black, 1930-39.

356,705,000 pounds and exports 203,828,000 pounds, both figures new records, with domestic deliveries showing the largest increase. (See fig. 2.)

Domestic consumption.—Domestic sales in 1939 (356,705,000 pounds) were 47 percent higher than in 1938 and 14 percent above the previous high of 1936. Reports from producers indicate that they were divided as follows: Rubber companies, 316,621,000 pounds (89 percent); ink companies, 21,929,000 pounds (6 percent); paint companies, 6,382,000 pounds (2 percent); and miscellaneous purposes, 11,773,000 pounds (3 percent). These data indicate that all four classes of consumption maintained their relative positions in 1939.

According to E. G. Holt, of the Bureau of Foreign and Domestic Commerce, who has again supplied data on rubber consumption, the total consumption of rubber in the United States reached the record-breaking total of 763,700 long tons, or 37 percent above the total of 558,800 tons consumed in 1938. Of the 1939 total, 592,000 tons was crude rubber, 170,000 tons reclaimed rubber, and 1,700 tons synthetic rubber. According to statistics of the Rubber Manufacturers Association 57,078,000 casings were produced in 1939 compared with the revised figure of 40,907,000 for 1938. Returns from the Federal excise tax on tires and tubes indicate that the total weight of casings sold in 1939 was 56 percent greater than in 1938. The production of camelpack for retreads has expanded rapidly in recent years and may have reached 50,000,000 pounds in 1939. This type of rubber utilizes more carbon black per pound of rubber than the average casing. As far as carbon-black consumption is concerned, these data indicate chiefly that an increase in the average size of casings outweighed a larger increase in the use of reclaimed (which uses relatively little carbon black) than in crude rubber.

The apparent world consumption of crude rubber was 1,090,000 long tons compared with the revised figure of 942,000 long tons in 1938. As the gain in consumption in the United States was somewhat larger than in the rest of the world, this country greatly strengthened its position as the leading rubber manufacturer.

The pick-up in business in 1939 was attended by a marked increase in advertising; consequently the apparent consumption of newsprint increased materially over 1938, though it fell far short of the 1937 total. According to data supplied by B. M. Frost, of the Bureau of Foreign and Domestic Commerce, the supply of newsprint available for domestic consumption in 1939 was 3,541,000 short tons compared with 3,089,100 in 1938. Sales of carbon black to ink companies increased 55 percent—from 14,131,000 pounds in 1938 to 21,929,000 in 1939. Possibly a higher percentage of carbon black is being used in inks, although probably further additions to manufacturers' stocks were made at the favorable prices.

Sales of carbon black to paint companies also rose materially—from 4,229,000 pounds in 1938 to 6,382,000 in 1939. According to E. C. Wood, of the Bureau of Foreign and Domestic Commerce, paint production in 1939 probably was about 15 percent higher than in 1938, with perhaps a much larger increase for black paints alone.

Sales of carbon black for miscellaneous purposes increased sharply; the 1939 total of 11,773,000 pounds was about 50 percent higher than in 1938 and slightly above the previous record (11,503,000 pounds) of 1937. No data are available as to which of the many miscellaneous uses expanded in 1939.

Exports and imports.[1]—Exports of carbon black passed the 200-million-pound mark for the first time in 1939, when the total was 203,828,000 pounds compared with 167,968,000 in 1938. Shipments to Germany declined in 1939, but this decrease was far outweighed by larger exports to British possessions.

The average export value continued to decline—4.36 cents per pound compared with 4.51 cents in 1938.

[1] Figures on exports and imports compiled by M. B. Price, of the Bureau of Mines, from records of the Bureau of Foreign and Domestic Commerce.

Exports of carbon black to the United Kingdom increased about 50 percent in 1939 to a total of 66,215,000 pounds. Exports to France (still second in importance) gained moderately in 1939, reaching 29,391,000 pounds, while those to Germany declined although the total (19,661,000 pounds) was third most important. There were notable increases in shipments to Brazil and China in 1939.

No particular trend was apparent in exports of carbon black by months, even after the war started. The highest monthly total was in April, the month of lowest exports in 1938. About 70 percent of the exports was shipped from the Galveston customs district, a slightly lower ratio than in 1938.

Carbon black exported from the United States, 1937–39, by countries

Country	1937		1938		1939	
	Pounds	Value	Pounds	Value	Pounds	Value
Argentina	3,115,630	$163,139	3,203,142	$151,669	4,234,248	$200,463
Australia	9,641,002	516,410	6,952,545	324,118	9,665,979	443,192
Belgium	5,164,255	234,743	5,459,202	250,475	4,650,082	212,169
Brazil	723,932	37,682	744,938	35,935	1,871,434	86,351
Canada	17,171,895	719,631	13,867,345	572,752	17,983,916	496,363
China	1,529,855	76,878	673,498	32,230	1,476,897	70,344
Czechoslovakia [1]	2,187,100	91,522	1,834,572	84,395	276,500	11,425
France	29,913,980	1,336,934	26,216,610	1,219,450	29,390,562	1,335,998
Germany [1]	27,441,114	1,172,649	23,646,635	1,076,568	19,660,905	857,907
India, British	1,002,210	44,196	949,455	44,340	2,703,106	123,530
Italy	6,956,079	300,972	9,764,699	440,881	8,260,261	375,258
Japan	11,929,498	619,790	9,172,849	443,483	10,617,734	482,538
Mexico	1,229,597	56,438	1,396,870	44,444	1,780,366	55,466
Netherland India	853,905	47,055	1,235,515	57,142	1,422,294	62,700
Netherlands	3,931,601	176,132	3,643,185	174,052	3,034,415	145,927
Norway	458,302	23,018	860,789	38,098	614,977	30,086
Poland and Danzig [1]	2,175,159	103,231	3,106,867	151,361	2,368,187	109,336
Spain	512,200	21,252	1,332,229	59,357	2,644,577	117,075
Sweden	1,549,753	86,637	2,714,415	130,063	4,435,409	213,570
Union of South Africa	3,155,311	138,449	1,792,986	75,375	4,222,092	186,958
United Kingdom	48,381,173	2,467,609	44,429,105	2,104,578	66,214,827	2,975,028
Yugoslavia	87,600	5,193	804,735	39,803		
Other countries	5,146,740	270,521	4,406,130	220,604	6,288,189	303,682
	194,252,882	8,700,082	167,968,316	7,579,883	203,827,817	8,888,666

[1] For statistical purposes trade with the Sudeten area, as far as ascertainable, is included with Germany, while trade with the other Czechoslovak provinces occupied by Germany, Hungary, and Poland has been included with these countries since March 15 or 19, 1939. After November 15, 1939, trade with Danzig and that part of Poland occupied by Germany has been included with Germany, and trade with that part of Poland occupied by the U. S. S. R. has been included with U. S. S. R.

Carbon black exported from the United States, 1938–39, by months and customs districts

Month	1938		1939	
	Pounds	Value	Pounds	Value
Jan.	14,869,135	$706,764	14,192,688	$612,887
Feb.	11,063,062	493,070	19,370,600	847,358
Mar.	14,814,670	654,988	19,935,052	874,362
Apr.	10,904,572	480,564	21,042,589	935,012
May	16,807,728	763,212	18,682,152	820,584
June	12,861,809	553,249	14,185,130	620,797
July	11,443,294	526,217	17,371,602	781,516
Aug.	12,421,213	536,079	13,505,738	579,400
Sept.	13,355,141	607,181	18,831,339	851,128
Oct.	16,639,788	776,318	17,300,319	737,795
Nov.	14,668,039	640,315	14,838,550	641,986
Dec.	18,100,880	781,926	14,670,978	636,771
	167,968,316	7,579,883	203,827,817	8,888,666

Customs district	1938		1939	
	Pounds	Value	Pounds	Value
Buffalo	89,711	$7,997	62,576	$3,055
Dakota	588,073	26,312	207,965	10,110
El Paso	1,344,900	26,617	1,649,717	46,882
Galveston	120,745,454	5,532,374	144,454,118	6,436,536
Los Angeles	349,312	14,496	1,074,945	40,332
Michigan	13,453,512	355,969	17,338,956	460,020
New Orleans	27,408,566	1,494,577	31,928,600	1,882,675
New York	112,410	36,746	208,276	26,888
Sabine	2,369,813	97,018	2,195,680	99,134
San Francisco	1,152,551	50,094	3,617,822	127,457
Vermont	122,555	4,469	289,240	7,285
Other districts	161,769	8,065	571,943	31,969
	167,968,316	7,579,883	203,827,817	8,888,666

Imports of "gas black and carbon black," as reported by the Bureau of Foreign and Domestic Commerce, totaled 6,901 pounds valued at $784 in 1939 compared with 390 pounds valued at $30 in 1938. Imports of acetylene black (all from Canada) nearly doubled in 1939, rising from 1,220,781 pounds valued at $127,889 in 1938 to 2,381,572 pounds valued at $248,332 in 1939.

STOCKS

The situation as to stocks of carbon black improved considerably during 1939; in place of a material increase, as in 1938, there was a moderate decline. Stocks on December 31, 1939 totaled 130,792,000 pounds, which was 35,367,000 pounds below stocks of January 1. In terms of quantity present stocks compare with those on hand during the early part of 1936 and in terms of days' supply, with those prevailing in the spring of 1937. Stocks in the form of finished rubber goods changed but slightly in 1939; stocks of casings held by manufacturers increased only from 8,451,000 on January 1, 1939, to 8,688,000 at the year end.

PRICES AND VALUES

In general, carbon-black prices in 1939 remained at the low levels reached in the early part of 1938. Although demand, both domestic and export, reached record levels, competitive conditions were such as to prevent any consistent advance.

The weighted average f. o. b. value at plants increased from 2.41 cents per pound in 1938 to 2.45 cents in 1939. This slight change is substantiated by the quotation for Zone A, the standard price in the industry, which has remained at 2.75 cents per pound since April 4, 1938.

In spite of the fact that exports expanded rapidly in the first 2 months of the war on a "seller's market," prices at the close of the year were slightly below those at the beginning. The average export value for 1939 was 4.36 cents per pound compared with 4.51 cents in 1938.

Quoted prices on various grades of carbon black, 1938–39, in cents per pound
[Oil, Paint, and Drug Reporter]

Date	Standard rubber, ink, and paint qualities (carlots)							Special grades for varnishes, lacquers, and enamels (cases delivered)						
	Zone[1]							Grade						
	A	B	C	D	E	F	G	1	2	3	4	5	6	7
1939:														
Jan. 1	2.75	3.10	3.19	3.25	3.41	3.75	3.00	3.75	8.50	13.00	27.50	40.00	60.00	110.00
Sept. 11	----	----	----	----	----	----	----	----	----	----	29.00	----	----	----
Average:														
1939	2.75	3.10	3.19	3.25	3.41	3.75	3.00	3.75	8.50	13.00	28.00	40.00	60.00	110.00
1938	2.75	3.09	3.20	3.24	3.40	3.73	3.22	4.04	8.50	13.00	27.50	40.00	60.00	110.00

[1] Zone A: Gulf coast ports: Galveston, Houston, Port Arthur, New Orleans, etc.; for coastwise delivery in North America.
Zone B: Arkansas, Colorado, Kansas, part of Missouri, New Mexico, and Texas except coastal ports.
Zone C: Pacific coast.
Zone D: Illinois, Iowa, and Wisconsin.
Zone E: Florida, Georgia, Indiana, Kentucky, Michigan, Ohio, Tennessee, West Virginia, and parts of New York and Pennsylvania.
Zone F: Atlantic seaboard States: Maine, Maryland, Massachusetts, New Hampshire, New Jersey, New York, North Carolina, parts of Pennsylvania, Rhode Island, South Carolina, Vermont, and Virginia.
Zone G: Mexico.

PROCEEDINGS IN THE UNITED STATES CIRCUIT COURT OF APPEALS FOR THE FOURTH CIRCUIT

No. 4837

BINNEY & SMITH COMPANY, Appellant,

versus

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Appellees

Appeal From the District Court of the United States for the Southern District of West Virginia, at Charleston

July 11, 1941, the transcript of record is filed and the cause docketed.

Same day, original exhibits are certified up.

Same day, order filing stipulation of counsel extending the time for thirty days from and after June 17, 1941, for the filing of the transcript of record, is filed.

July 15, 1941, the appearance of Dean S. Edmonds, Roger T. McLean and Howard R. Klostermeyer is entered for the appellant.

July 21, 1941, the appearance of George P. Dike, Arthur M. Smith and Osman E. Swartz is entered for the appellees.

September 13, 1941, petition of appellant to continue case from the October term to the November term, 1941, is filed.

Same day, stipulation and consent to a continuance is filed.

ORDER CONTINUING CASE—Filed September 17, 1941

[Style of Court and Title Omitted]

Upon the Joint Application of the parties, by their respective counsel,

It Is Ordered that the above-entitled cause be, and the same is hereby, continued from the October term, 1941, to the November term, 1941, of this Court.

It Is Further Ordered that the brief and appendix for the appellant be filed on or before October 10, 1941, and that

the brief and appendix for the appellees be filed on or before October 25, 1941.

September 15, 1941.

John J. Parker, Senior Circuit Judge.

November 7, 1941, brief on behalf of appellees is filed.
November 8, 1941, stipulation as to briefs is filed.
Same day, brief on behalf of appellant is filed.
Same day, joint appendix to the respective briefs is filed.
November 12, 1941, reply brief on behalf of appellant is filed.

ARGUMENT OF CAUSE

November 14, 1941, (November term, 1941) cause came on to be heard before Parker, Soper and Dobie, Circuit Judges, and was argued by counsel and submitted.
Same day, the original physical exhibits lodged by counsel.

OPINION—Filed January 6, 1942.

UNITED STATES CIRCUIT COURT OF APPEALS
FOURTH CIRCUIT

No. 4837.

BINNEY & SMITH COMPANY, Appellant,

versus

UNITED CARBON COMPANY AND UNITED CARBON COMPANY, INC., Appellees

Appeal from the District Court of the United States for the Southern District of West Virginia, at Charleston.

(Argued November 14, 1941. Decided January 6, 1942.)

Before PARKER, SOPER and DOBIE, Circuit Judges.

Dean S. Edmonds (Roger T. McLean; Howard R. Klostermeyer; Price, Smith & Spilman, and Pennie, Davis, Marvin & Edmonds on brief) for Appellant, and George P. Dike (Arthur M. Smith and Osman E. Swartz on brief) for Appellees.

PARKER, Circuit Judge:

This is a suit for infringement of Wiegand and Venuto Patent No. 1,889,429, covering carbon black pellets and a process for making them. It was instituted by Binney & Smith Company, assignee of the patent, against the United Carbon Company and the United Carbon Company, Inc. Only the product claims of the patent, claims 1 and 2, are in suit. The court below held these claims invalid as lacking novelty and invention and also because of indefiniteness in the description of the product. The court held further that, if broadly construed, the claims would be infringed by the product of defendants, but that, even if valid, they should be strictly construed and that, when so construed, they were not infringed. From an order dismissing the suit, the plaintiff has appealed.

We think that the evidence shows conclusively that the patentees under the patent in suit were the first to solve the problem presented by the dust nuisance arising from the use of carbon black in the manufacture of rubber tires for motor vehicles. Carbon black is produced by burning low quality gas and capturing the unconsumed carbon carried in the flames. As it comes from the burning house, this is a loose, light, fluffy product, weighing about three pounds to the cubic foot. Through agitation and compression, the air is driven out of it to as great extent as possible; but even after this is done, the product is a finely divided powder, weighing only about twelve pounds to the cubic foot and composed of particles a millionth of an inch in diameter, so small that they can be seen only with the aid of an electron microscope. By 1915 it was recognized that the use of carbon black as a filler in the manufacture of rubber for automobile tires greatly improved the quality of the tires; but the dust arising from the use of the extremely fine powder in the process of manufacture was all but an intolerable nuisance. Efforts to produce carbon black in a form which would eliminate the dust nuisance engaged the attention of a number of the best minds connected with the rubber industry, but until the issuance of the patent in suit these efforts were unavailing. A large number of patents were applied for and issued, but none covered a product which would meet the demands of the industry. Some attempted granulation of the powder by the use of binders such a soil or

tar, but these failed because the industry objected to the binders which they contained. Others attempted wetting the carbon black with water or other volatile liquid and then drying it out, but these failed because the result was a dried "mud cake" the breaking up of which revived the dust nuisance, and the particles of which were "harsh" and did not disperse satisfactorily in the rubber. None succeeded in putting the carbon black in a form which the industry was willing to accept.

The invention of plaintiff's patent consisted in producing carbon black in the form of small rounded pellets, which were dustless and which would disintegrate into the original small particles of carbon black when subjected to the pressure involved in the rubber manufacturing process. These pellets could be easily handled and could be transported in tank cars or stored in bulk instead of being shipped in paper bags, as required in the case of the carbon black powder. They were made by wetting carbon black with water, introducing into the mass a liquid such as gasoline, and then subjecting the mass to violent churning or agitation, with the result that the carbon particles would agglomerate in the form of small rounded pellets, after which the gasoline would be evaporated, leaving the pellets in the pure carbon state. The process is thus described in the specification of the patent:

"The carbon black is first mixed with water so as to form a comparatively thin paste with most of the occluded gases driven out. After thorough mixing gasoline is added and the whole mass thoroughly agitated. As a result substantially all of the carbon black floats on top of the water in the form of pellets and may be readily removed by filtering, decanting or the like. These pellets may then be dried to remove the absorbed gasoline and such moisture as there may be contained therein. • • •

"On the first addition of gasoline to the black and water paste, with agitation, the mass seems to thicken like an emulsion; then on further shaking, it seems to flocculate into granules; as the agitation goes on, a slight creaming to the top of the pellets or a scum takes place, then gradually the pellets form and float to the top. The end point is reached when the water below is free of carbon-clear.

"The pellets are then screened off by any of the various methods, placed in an oven or a recovery apparatus and freed from solvent and water. * * *

"Merely as an example of the relative proportions which we have found to operate satisfactorily, we give the following by weight:

	Parts
Carbon	5
Water	40 to 80
Gasoline	7 to 9

"In carrying out our improved process with these proportions, the mass may be thoroughly shaken or otherwise agitated for about five minutes and the resulting pellets will be of about one-sixteenth of an inch in diameter and will be fairly uniform in size.

"The pellets are very porous, of substantially spherical or globular form, have a smooth somewhat lustrous outer surface which is not easily broken by handling, are more compact than untreated carbon, are fragile under light pressure, and may be easily reduced to soft minute particles which cannot be told from the original particles except that possibly they have a more unctuous feel. They somewhat resemble lead shot and may be rolled in the hand without dirtying or dusting. Apparently the outer surface portion or shell of each pellet is slightly more compact than the inner part, but still porous."

Claims one and two of the patent, being the product claims here relied on, are as follows:

"1. Substantially pure carbon black in the form of commercially uniform, comparatively small, rounded, smooth aggregates having a spongy porous interior.

"2. As an article of manufacture, a pellet of approximately one-sixteenth of an inch in diameter and formed of a porous mass of substantially pure carbon black."

The evidence leaves no doubt but that the dust problem in the rubber industry was solved by the pellet produced by the patent. Rubber manufacturers were wary of accepting it at first; and its acceptance was further retarded by reason of a higher price being demanded for it than was charged for the carbon black powder, but the evidence

leaves no doubt but that it was regarded from the beginning as a valuable improvement and was destined to have wide commercial success. Application for the patent was not filed until 1927. In 1928 sample lots were sent out to prospective customers. In 1929 there were commercial sales of 20,000 pounds, in 1930 of 164,000 pounds, in 1931 of 194,000 pounds and in 1932 of 281,000 pounds. In 1932 there was discovered an improvement in the process of manufacture which, by eliminating the use of expensive gasoline, cheapened the process and resulted, we think, in an improvement in the product. From then on the sales of plaintiff steadily mounted, being 800,000 pounds in 1933, 3,300,000 pounds in 1934, 30,000,000 pounds in 1936 and 97,000,000 pounds in 1939. Other companies in the meantime had entered into the production of the pelleted product, so that by 1939 this form of carbon black had virtually superseded the powdered form for use in the rubber industry.

With reference to improvements in the process and product, these have resulted from the use of only one liquid and, finally, no liquid at all in the formation of the pellets. It will be observed that the process of plaintiff's patent prescribes churning or agitation of the mass of carbon black and liquids. This churning or agitation is evidently the really important thing in the process, and, when properly carried on, is what causes the carbon black to agglomerate into pellets, and to do so whether any liquid is present or not. These one liquid and no liquid processes, being different from the processes of plaintiff's patent, were of course patentable, and the use of such processes, omitting as they did elements essential to plaintiff's process, did not infringe that process; but this does not mean that the product produced by them, even if an improvement on the product of the patent in suit, does not infringe the product claims of that patent.

The first question which arises with respect to the product claims in suit is whether such a product was patentable, not being a "combination of matter", but being composed of pure carbon black. We think that it was. It was patentable as a "manufacture". As said by Judge Acheson in *Johnson* v. *Johnston*, 60 F. 618, 620: "The term 'manufacture', as used in the patent law, has a very comprehensive sense, embracing whatever is made by the art or industry of man, not being a machine, a composition of

matter or a design. Curt. Pat. sec. 27; 1 Rob. Pat. sec. 183." In *J. E. Baker Co.* v. *Kennedy Co. et al.*, 3 Cir. 253 F. 739, a patent was sustained covering magdolite, a product which was no more than dolomite rock with certain objectionable features removed. And in *Union Carbide Co.* v. *American Carbide Co.*, 2 Cir. 181 F. 104, it was held that calcium carbide in the form of crystals was patentable as a new product, although the chemical combination in other forms was well known. And there can be no question as to the patentability of the product as distinguished from the process for making it, which was also patentable. *Rubber Co.* v. *Goodyear*, 9 Wall. 788, 796; *Leeds & Cotten* v. *Victor Talking Mach. Co.*, 213 U. S. 301, 319; *Hide-Ite Leather Co.* v. *Fiber Products Co.*, 1 Cir. 226 F. 34, 36. As said in the case last cited:

"It is a well-recognised rule that a new product, having definite characteristics by which it may be identified, and which distinguish it apart from the process by which it was produced, may be properly described by such characteristics, and when so claimed and described the claims are not limited to the process. Maurer v. Dickerson, 113 F. 870, 51 C. C. A. 494."

We entertain no doubt as to the patentability of the product here involved. What we have is not an obvious change of form which anyone could make, once the desirability of such change was recognized, as was the breaking up of the flakes of glue in *Glue Co.* v. *Upton*, 97 U. S. 3, but the manufacture of an entirely new form of a product, having new characteristics and advantages, and solving problems in connection with the use of the product which had long baffled those most highly skilled in the art. Although a form of carbon black in which the dust nuisance would be eliminated had been sought diligently, it had not been found up to that time. The product of the patent solved not only this problem but also problems connected with handling and transportation. It did this by agglomerating the tiny particles of the powdered product into small granules or pellets which were dustless, which were flowable and which would disintegrate in the process of rubber manufacture so as to preserve the advantages of the dispersability of the tiny particles. Carbon black in such form was a new and highly useful product; and we see no reason why those who first made it should not be protected with

respect to the new product which they had brought forth as well as with respect to the process which they had devised for making it.

We have carefully considered the patents relied on as anticipations, and we do not think that they anticipate. Thirty-eight prior patents were introduced in evidence in the court below, which, in itself, tends to weaken the contention as to lack of novelty. As we said in *Hoeltke* v. *C. M. Kemp Mfg. Co.*, 4 Cir. 80 F. 2d 912, 917: "Such a citation of so many prior patents almost inevitably means either that none of them is nearly like the invention of the patentee and that the attempt is being made to invalidate the patent because the patentee has brought together for the purposes of his invention devices to be found in prior patents of different character, or that prior attempts to solve the problem with which he was confronted have not met with success." Seven of these prior patents were cited by the court below but only five of them are relied upon by defendants in their brief in this court, viz.: Geer No. 1,245,700 (1917), Lewis No. 1,263,082 (1918), Knowlton and Hoffman No. 1,286,024 (1918), Trent British Patent No. 183,430 (1923), and Coffin and Keen No. 1,561,971 (1925).

None of these patents discloses any such product as is covered by the patent sued on. The Geer patent involves the use of water or gasoline to agglomerate the carbon black particles and the use of the resulting product before it has dried out, with the water or gasoline acting as a binder for the particles as they go into the rubber batch. The patent was a failure because the use of a binder of any sort was objectionable to the industry. The Lewis patent involves the caking of carbon black by mixing it with a liquid and then drying the mixture; but this does not produce the granules or pellets of the patent but a cake which has to be broken up, with a revival of the dust nuisance. The Knowlton and Hoffman patent speaks of the granulation of the carbon black by mixing it with a volatile liquid and then drying it; but it does not disclose how this is to be done and the only illustration of the process which is given requires the use of starch as a binder. Nothing was contributed to the art by any of these patents. They were granted to technologists of recognized eminence in the industry; and it is a safe assumption that, if they had contributed anything to the solution of the problem

which confronted the industry, their value would have been recognized and they would have been used. As it is, they show merely that the industry knew what was needed, but not how to achieve it.

The Trent British patent relates to a process for agglomerating coal dust with a binder of fuel oil to produce a fuel. The patent is broad enough to cover the use of carbon black, as the term "carbonaceous material" is used to describe the dust agglomerated; but the binder condemns the product for use in the rubber industry and differentiates it from the product of the patent in suit. The Coffin and Keen patent relates to a process for drying clay or "other pulverulent material". While one of the claims is for "a fluent dried pulverulent inorganic material in the form of very small dried friable globular masses", it nowhere discloses how to make these successfully of carbon black; and the fact that it seems to have occurred to no one to use the process disclosed to produce something which the industry needed so badly is a practical answer to the contention that it anticipates the patent in suit. The high temperature prescribed by the patent for use in the drying process would preclude its use in connection with carbon black.

We see no merit in the contention that the product claims are vague or indefinite. They describe the product, not in terms of function as defendants contend, but in terms of size, content, shape and texture. The pellets are described as small aggregates composed of substantially pure carbon black, as being rounded and smooth in shape, and as having a spongy, porous interior. We think that this describes them as accurately as they can be described. There is no requirement, in law or in common sense, that in the description the exact size of the pellets be given, if this were possible, or that the exact nature of the porosity or friability be set forth. The patentees had made a substantial contribution to an important art by solving a problem which had baffled other technological experts who had addressed themselves to it. They described their product in terms by which any one could identify it; and they set forth a process by which it could be manufactured. Theirs was a pioneer patent as applied to the particular art; and its claims are entitled to a liberal construction. Walker on Patents 6th ed. sec. 232. Greater

precision or particularity in the description of the pellets might have had the effect of limiting the scope of the patent to a narrow section of the field which patentees were entitled to occupy.

Nor are we impressed with the contention that the claims covering this new and useful product are to be given a narrow or limited construction because other broad claims were disallowed by the patent office and later abandoned. The rule is well settled, of course, that claims allowed may not be construed so as to cover broader claims which have been rejected and abandoned. *Doughnut Machine Corp.* v. *Joe Lowe Corp, et al.*, 4 Cir. 67 F. 2d 135, 138. This does not mean, however, that the language of a claim is to be denied its ordinary meaning because another claim containing somewhat similar language has been rejected. The abandoned claims were rejected because "so broad that the product might be quite different" from that of patentees and as not properly defining appellant's invention. Whether they were thought broad enough to include a pellet made with a binder or not, certain it is that the claims allowed adequately describe the product, which should not be denied protection because other and broader claims, thought not to describe it adequately, have been rejected. It is worthy of note that none of the citations upon which the rejections were based disclosed anything like the product of the patent.

On the question of infringement, we think there can be no reasonable doubt. The court below held that plaintiff's product and defendant's product "are generally similar" and that the claims in suit would be infringed if construed broadly. Since the patent was for a pioneer invention in this particular art, we think, as stated above, that the claims are entitled to a broad and liberal construction. Irrespective of this, however, we think that there is infringement by defendant's product for the reason that it is substantially the same thing as the product of the patent. There are the same small pellets of carbon black, which are substantially pure for the practical purposes of the art. They are smooth, round and flowable, just as are the pellets of the patent. They are substantially the same size, and are porous and friable.

If, as is argued, the improved processes followed by defendants result in firmer pellets more easily handled, this does not avoid infringement, if the product is substan-

tially the same as that covered by the patent; for nothing is better settled than that improvement does not avoid infringement. Walker on Patents 6th ed. sec. 452 and cases cited. The improver is entitled to protection by patent, just as is the original inventor; but this does not entitle him to use the original invention. The monopolies of the two patents are mutually exclusive. The improver may not make the product even by his improved process without license from the original inventor; and the original inventor may not make the improved product, or use the improved process, without license from the improver. If this were not so, the value of patents for the most useful inventions would be swept away by any improvements made on them, in the one case, and the granting of a patent would freeze the state of the art, in the other. As it is, each is allowed a monopoly for the statutory period on what he has invented and no more.

For the reasons stated, we are of opinion that the product claims sued on are valid and infringed by the product of defendants. The decree appealed from will accordingly be reversed and the case will be remanded for further proceedings not inconsistent herewith.

Reversed.

DECREE—Filed and Entered January 6, 1942

UNITED STATES CIRCUIT COURT OF APPEALS, FOURTH CIRCUIT

No. 4837

BINNEY & SMITH COMPANY, Appellant,

vs.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Appellees

Appeal from the District Court of the United States for the Southern District of West Virginia

This Cause came on to be heard on the transcript of the record from the District Court of the United States for the Southern District of West Virginia, and was argued by counsel.

On Consideration Whereof, It is now here ordered, adjudged, and decreed by this Court that the decree of the

said District Court appealed from, in this cause, be, and the same is hereby, reversed with costs; and that this cause be, and the same is hereby, remanded to the District Court of the United States for the Southern District of West Virginia, at Charleston, for further proceedings in accordance with the opinion of the Court filed herein.

January 6th, 1942.

John J. Parker, Senior Circuit Judge.

January 22, 1942, petition of appellees for an extension of time to present petition for rehearing and for stay of mandate is filed.

ORDER EXTENDING TIME TO PRESENT PETITION FOR REHEARING AND STAYING MANDATE—Filed January 24, 1942

(Style of Court and Title Omitted)

Upon the Application of the Appellees, and for good cause shown,

It Is Ordered by this Court that the time within which the Appellees may present petition for rehearing in the above case be, and the same is hereby, extended to and including February 20, 1942, and

It Is Further Ordered that the mandate of this Court in this case be, and the same is hereby, stayed pending the action of the Court on the petition for rehearing, provided said petition for rehearing is presented on or before February 20, 1942.

January 23, 1942.

John J. Parker, Senior Circuit Judge.

February 5, 1942, petition of appellees for a further extension of time to present petition for rehearing and for stay of mandate filed.

ORDER FURTHER EXTENDING TIME TO PRESENT PETITION FOR REHEARING AND STAYING MANDATE -Filed February 10, 1942

(Style of Court and Title Omitted)

Upon the Application of the Appellees, and for good cause shown,

It Is Ordered by this Court that the time within which the Appellees may present petition for rehearing in the above case be, and the same is hereby, extended to and including March 7, 1942.

It Is Further Ordered that the mandate of this Court in this case be, and the same is hereby, stayed pending the action of the Court on the petition for rehearing, provided said petition for rehearing is presented on or before March 7, 1942.

February 9, 1942.

Armistead M. Dobie, U. S. Circuit Judge.

PETITION OF APPELLEES FOR REHEARING—Presented February 23, 1942

UNITED STATES CIRCUIT COURT OF APPEALS FOR THE FOURTH CIRCUIT

No. 4837

BINNEY & SMITH COMPANY, Plaintiff-Appellant,

v.

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Defendants-Appellees

PETITION FOR REHEARING

Your petitioners, United Carbon Company and United Carbon Company, Inc., defendants-appellees, respectfully pray that the decision in the above-entitled action be reconsidered, that the case be reheard, and that the mandate be stayed pending the decision, for the reason that:

The Court overlooked the fact that the prior patent of Knowlton & Hoffman does tell how to make the product of the claims in suit. The instruction of that patent is:

(1) Mix enough water with the gas-black to render the gas-black dustless, yet not pasty;

(2) granulate the mix in a closed vessel preferably containing rapidly-revolving paddles;

(3) dry the resulting grains.

Thereby, Knowlton & Hoffman produce "friable grains or small lumps, dry and substantially free from dust", like

the product of defendants' Sayre process which the Court held to be the product of the claims in suit.

United Carbon Company, United Carbon Company, Inc., by Osman E. Swartz, Attorney.

We, the counsel for defendants-Appellees in the above-entitled action, hereby certify that the foregoing petition is presented in good faith and not for delay.

Hugh M. Morris, George P. Dike, Osman E. Swartz, Arthur M. Smith, Counsel for Defendants-Appellees.

PER CURIAM OPINION ON PETITION FOR REHEARING—Filed March 11, 1942

UNITED STATES CIRCUIT COURT OF APPEALS, FOURTH CIRCUIT

No. 4837

BINNEY & SMITH COMPANY, Appellant,

versus

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC., Appellees

Appeal from the District Court of the United States for the Southern District of West Virginia, at Charleston

On Petition for Rehearing

(Petition presented February 23,1942. Decided March 11, 1942)

Before Parker, Soper and Dobie, Circuit Judges

Dean S. Edmonds (Roger T. McLean; Howard R. Klostermeyer; Price, Smith & Spilman, and Pennie, Davis, Marvin & Edmonds on brief) for Appellant, and George P. Dike (Arthur M. Smith and Osman E. Swartz on brief) for Appellees

PER CURIAM:

The petition for rehearing herein presents no matter not already fully considered. Nothing in the Knowlton and Hoffman patent has been overlooked. It is true that

that patent speaks of using "water or other volatile liquid alone"; but the patent does not disclose how this may be done in such way as to produce the pellets of the patent in suit, and the mere reference to the Geer patent did not enable the industry to combine the two processes so as to produce them. The use of the Knowlton and Hoffman process without the starch binder results in an agglomerated product in the form of a mud cake which is broken up with the revival of the dust nuisance into particles, which because of their harshness, are not readily dispersible in the rubber. As pointed out in the original opinion, nothing was contributed to the art either by this patent or by the patent to Geer. Both were mere failures in the attempt to solve the problem which was solved by the product of the patent in suit.

Petition Denied.

ORDER DENYING REHEARING—Filed and Entered March 11, 1942

(Style of Court and Title Omitted)

This Court having at its January term, 1942, rendered its decision reversing the decree of the said District Court appealed from in this cause, and the appellees having on February 23,1942, presented to the Court a petition for a rehearing of the said cause, and the same having been carefully considered,

It Is Now Here Ordered By This Court that the rehearing asked for, be, and the same is hereby, denied. Let mandate issue after the expiration of five days.

March 11, 1942.

John J. Parker, Senior Circuit Judge.

March 13, 1942, petition of appellees for stay of mandate pending application for writ of certiorari is filed.

ORDER STAYING MANDATE PENDING APPLICATION FOR WRIT OF CERTIORARI—Filed March 13, 1942

(Style of Court and Title Omitted)

Upon the Application of the Appellees, by their counsel, George P. Dike, and for good cause shown,

It Is Ordered that the mandate of this Court in the above entitled cause be, and the same is hereby, stayed pending

the application of the said Appellees in the Supreme Court of the United States for a writ of certiorari to this Court, unless otherwise ordered by this or the said Supreme Court, and provided said application is filed in the said Supreme Court within 30 days from this date.

March 13, 1942.

John J. Parker, Senior Circuit Judge.

CLERK'S CERTIFICATE

UNITED STATES OF AMERICA,
Fourth Circuit, ss:

I, Claude M. Dean, Clerk of the United States Circuit Court of Appeals for the Fourth Circuit, do certify that the foregoing is a true copy of the joint appendix to the briefs of the respective parties, and the proceedings in said Circuit Court of Appeals in the therein entitled cause, as the same remain upon the records and files of the said Circuit Court of Appeals, and constitute and is a true transcript of the record and proceedings in the said Circuit Court of Appeals in said cause, made up in accordance with the direction of counsel for the appellees, for use in the Supreme Court of the United States on an application for a writ of certiorari.

In Testimony Whereof, I hereto set my hand and affix the seal of the said United States Circuit Court of Appeals for the Fourth Circuit at Richmond, Virginia, this 23rd day of March, A. D., 1942.

Claude M. Dean, Clerk, U. S. Circuit Court of Appeals, Fourth Circuit. (Seal.)

SUPREME COURT OF THE UNITED STATES

No. —

UNITED CARBON COMPANY and UNITED CARBON COMPANY, INC.,
Petitioners,

v.

BINNEY & SMITH COMPANY, Respondent

STIPULATION AS TO PRINTING RECORD

It is hereby stipulated and agreed by and between counsel for the respective parties hereto that the following papers

included in the record heretofore certified to the Supreme Court of the United States by the Clerk of the Circuit Court of Appeals for the Fourth Circuit and in addition to the Appendix (Joint) heretofore printed by the parties shall be printed for presentation to the Court, together with the said Appendix (Joint):

(1) Pleadings;

(2) Defendants' Exhibit No. 1—Banbury Mixer Bulletin No. 171;

(3) Defendants' Exhibit No. 32—Printed Circular of Tamms Paint Mixer;

(4) The following patents from defendants' Exhibit No. 22:

No. 207013—Carter
No. 671804—Metcalf
No. 151236—Trent British.

(5) The following patents from defendants' Exhibit No. 23:

No. 1076235—Rodman
No. 1239221—Rodman
No. 1239368—duPont
No. 1466571—Thorssell and Troell.

(6) Plaintiff's Exhibit No. 19—Minerals Year Book 1940, Front Cover and Pages 1 to 8;

(7) Opinion of the Circuit Court of Appeals filed January 6, 1942;

(8) Defendants-Appellees' petition for rehearing;

(9) Per Curiam opinion of Circuit Court of Appeals denying petition for rehearing, dated March 11, 1942;

(10) Judgment of the Circuit Court of Appeals;

(11) Order staying mandate;

(12) This stipulation;

(13) Such other matters as the Rules of the Supreme Court of the United States requires to be certified.

United Carbon Company and United Carbon Company, Inc., by George P. Dike, Its Attorney. Binney & Smith Company, by Dean S. Edmonds, Its Attorney.

[Endorsed:] In the Supreme Court of the United States. United Carbon Company and United Carbon Company, Inc., Petitioners, vs. Binney & Smith Company, Respondent. Stipulation as to Printing Record. Hugh M. Morris, Attorney at Law, Wilmington, Delaware.

(9682)

[fol. 499] SUPREME COURT OF THE UNITED STATES

ORDER ALLOWING CERTIORARI—Filed May 11, 1942

The petition herein for a writ of certiorari to the United States Circuit Court of Appeals for the Fourth Circuit is granted.

And it is further ordered that the duly certified copy of the transcript of the proceedings below which accompanied the petition shall be treated as though filed in response to such writ.

[fol. 500] SUPREME COURT OF THE UNITED STATES

STIPULATION AS TO PRINTED RECORD—Filed May 30, 1942

A writ of certiorari having been granted in this cause, it is hereby stipulated by and between counsel for the respective parties that the record to be printed, under rule 38 subdivisions 7 and 8 of the Rules of the Supreme Court of the United States, for the use of the Court and counsel in said cause, shall be the same record as that which was presented with the petition for writ of certiorari under stipulation of the parties as to printing the record theretofore filed with the Clerk of the Supreme Court of the United States in said cause, with the exceptions:

(1) That item No. (6) in said stipulation, which was incorrectly stated as "Plaintiff's Exhibit No. 19—Minerals Year Book 1940, Front Cover and Pages 1 to 8", be changed to read as follows:

"Plaintiff's Exhibit No. 8—Minerals Year Book 1940, Front Cover and Pages 1 to 8";

and that such Minerals Year Book 1940 be substituted for [fol. 501] Minerals Year Book 1939 appearing at pages 471 to 480, inclusive, in that part of the record heretofore printed in this Court.

(2) That, inasmuch as the Clerk of the Supreme Court of the United States has heretofore been supplied with nine copies of defendants' Exhibit No. 1—Banbury Mixer Bul-

letin No. 171, which copies will be available for use in making up a similar number of copies of the printed record pursuant to this stipulation for the use of the Court, and inasmuch as no further copies of said exhibit can be procured and the cost of reproducing the same would be very substantial, said exhibit need not be included in the additional copies of the printed record to be supplied under rule 38(7) of the Rules of the Supreme Court of the United States, in this cause.

May —, 1942.

Hugh M. Morris, Counsel for Petitioners. Wm. S. Edwards, Counsel for Respondent.

[fol. 502] [File endorsement omitted.]